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DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

George K. Burgess, Director

CIRCULAR OF THE BUREAU OF STANDARDS, No. 145

**SUMMARY OF TECHNICAL METHODS
FOR THE UTILIZATION OF MOLASSES**

COLLATED FROM PATENT LITERATURE

January 28, 1924



PRICE, 15 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
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ABSTRACT.

Economic conditions the world over have made it generally recognized that the future of the American beet-sugar industry is to a great extent dependent on the profitable utilization of the molasses produced. The latter contains numerous valuable substances which have never been successfully recovered outside of Germany. For many years that country has seen fit to veil its developments and discoveries and to maintain the strictest secrecy regarding the operation of its molasses plants. The scientific literature on the subject is practically barren, so far as the actual results achieved in Germany are concerned. In order to carry out the necessary experimental work for the American industry every possible source of information has been investigated. After a few clues were obtained the United States Patent Office literature was searched and eventually over 1,000 German patents on molasses utilization and associated subjects were uncovered. The difficulty in locating these patents was due to the fact that they are not listed under sugar or molasses or subjects that were known to the bureau's investigators. The value of the information contained in many of these patents was so apparent and the necessity for knowledge of their contents was so obvious that the present résumé of the more important patents on the recovery of the nonsugars has been compiled.

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I. INTRODUCTION.

(a) **ECONOMIC CONDITIONS.**—Economic conditions the world over, especially in the older and basic industries, have made it imperative that each industry utilize its raw materials to the fullest possible degree. This fact is particularly applicable to the American beet-sugar industry, and the necessity for a more efficient utilization of the beet molasses produced in the United States has long been recognized. Perhaps no other commercial commodity produced on a large scale has had as wide a variation in actual value. At times it has been a valuable asset to the companies producing it, and at times it has been an actual liability. It contains numerous valuable substances, and probably all the American beet-sugar producing companies have at some time given serious consideration to the development of economically successful methods for the recovery of these substances. Sufficient has been accomplished in this direction to prove that the problems involved are highly scientific and technical, and that the information desired can be secured only at the expense of long-continued research in specially designed laboratories.

(b) **FOREIGN PRACTICE.**—It has become generally recognized that the future of the American beet-sugar industry is in no small degree dependent on the profitable recovery of these by-products. Naturally, it is to be expected that the greatest development in this field should be attained by European beet-sugar companies where the industry was first developed. Prior to 1914 approximately one-half the world's sugar supply was produced from sugar beets, and, with the economic conditions

which have long prevailed in Europe, it is logical to expect that all the resources of modern science would have been utilized long ago to develop methods of recovery of the valuable by-products known to exist in such a large-scale production commodity as beet molasses. Much valuable research work has been done to that end in various interested countries, particularly France and Germany. It is in the latter country, however, judging from all available information, that great success has been attained. Four beet-sugar utilization plants were in operation in that country until some time between 1914 and 1918, when two of them were destroyed or converted to other purposes made necessary by the stress of war. Owing to the necessity of harvesting the sugar beet and recovering the sugar stored in it within a comparatively short time, it is impracticable to operate beet-sugar plants the entire year. The discovery of scientific methods of recovering the by-products made it possible to design the molasses-utilization plants to operate 12 months in the year. The great economy in operation which results is evident. The most noted of the four German plants is known as the Dessau plant, located in the city of Dessau.

Unfortunately for the world in general, the German beet industry has seen fit to veil its developments and discoveries in this field and to maintain the strictest secrecy regarding the operation of its plants. The scientific literature of the beet-sugar industry is practically barren so far as the actual results achieved in Germany are concerned. In the Bureau of Standards' efforts to collect existing information recourse was had to the U. S. Patent Office files in a search which led eventually to the uncovering of over 1,000 German patents on molasses utilization and associated subjects. The value of the information contained in many of these patents was so apparent, and the necessity for a knowledge of their contents was so obvious, especially in view of carrying out experimental work on American beet molasses, that the necessity for a complete patent survey was at once evident.

(c) SCOPE OF PATENT SEARCH.—A search has been carried on through the files of the patents of the United States, Great Britain, Germany, and France as found at the United States Patent Office in Washington, supplemented by references to and abstracts of patents found in the current literature. Considerable difficulty was experienced in making the search as comprehensive as possible because of the large number of classifications it was necessary to cover, especially in the German files, in order to locate not only

the most direct but the related patents concerned. It must be kept in mind that under the present systems of classification in vogue in the various countries it is impossible to choose only those patents which bear directly upon the desired subject. Neither is it possible to know which, by virtue of their titles alone or even their texts, are connected with that subject while appearing in an entirely different section of the patent classification. There remains also the fact that in many cases, contrary to the spirit of the patent laws, the titles and texts of the patents are so framed as to deliberately camouflage the nature and purpose of the process described. These conditions make it exceedingly difficult to select, without fail, all patents connected with a given problem and make it appear possible that some may have been overlooked which might profitably have been included. In presenting the following survey, therefore, no claim is made as to its completeness, but we simply state that the patents listed include all those which seemed to bear directly on the problem at hand.

(d) **METHODS OF UTILIZING WASTE MOLASSES.**—At the beginning of the search into the problem of the utilization of waste molasses, it is necessary to define the scope of the field to be covered. Waste molasses has found many uses, more or less practical. For instance, we find it being used as a fertilizer, a cattle feed, a source of alcohol, a medium for the production of yeast, a fuel, a filler in many manufactured products, and, in Germany, a source of potash and various valuable organic and inorganic chemicals. It would have proven very difficult to have covered adequately the patents in connection with all these various uses, and it was, therefore, necessary to limit the scope of the investigation in order to utilize the limited time to the greatest advantage.

(e) **BASIS OF PATENT SURVEY.**—Certain assumptions were made as to the nature of molasses and the practical forms of utilization as applied to the conditions in the United States. In the first place, greatest stress was laid on the discard molasses and waste water from the beet-sugar industry. Since the molasses from this source is not suitable for table use, it must be utilized in other ways. It was assumed, moreover, that in the modern sugar house the maximum quantity of available sucrose is extracted from the molasses by a suitable saccharate process, such as Steffen's process. This naturally eliminates from the discussion all consideration of the various saccharate processes, a subject which is of great importance and which we expect to discuss in a later publication. The residue left after the application of a saccharate process is a

waste water which contains all the nonsugars, consisting chiefly of inorganic salts of potassium and sodium and various organic nitrogenous bodies. In applying the Steffen cold saccharate process in most sections of the beet-sugar area of the country it has been found necessary to discard a portion of the molasses without desugarizing in order to eliminate from the extraction system certain harmful impurities which accumulate by recirculation. This discard molasses, of course, carries the same kinds of nonsugars, and, in addition, a relatively much larger amount of sugar.

The use of molasses as a source of alcohol and yeast is quite well known and generally practiced, but the erection of a plant for this purpose is expensive. In European countries beet sugar has long been the basic raw material for much of the alcohol produced. In the United States, however, grains as well as molasses have been utilized for the production of a considerable quantity of alcohol. More recently the marked decline in the price of molasses has resulted in its greatly increased use for this purpose. Recently there have been promising developments in the manufacture and use of alcohol as a motor fuel. Anhydrous ethyl alcohol is being produced in considerable quantities. Recent tests at this bureau have shown that a motor fuel consisting of 30 per cent of this material and 70 per cent gasoline gives results superior to gasoline. It is believed that the recent accomplishment of producing anhydrous ethyl alcohol in large quantities at a cost not greatly in excess of that of gasoline will prove most significant in the solution of the motor-fuel problem. Having utilized the discard molasses in this manner, however, we should still have to dispose of the valuable nonsugars now contained in the two residual liquors; first, the saccharate waste water mentioned above, and, second, the distillery slops or vinasses as they are called. The problem of the recovery of these substances in economically valuable forms again presents itself, whether the sugar of the molasses is recovered by a saccharate process or is converted to alcohol and yeast.

Of the other uses mentioned, disposal of the molasses as a fertilizer, a feed, or as a filler in various industrial products, presents a limited outlet and a poor financial return, although these methods serve their purpose in the agricultural economy.

The final consideration in any case in deciding upon the most practical plan for the utilization of waste molasses must hinge upon the economic or financial aspects of the problem. These facts naturally lead us to a consideration of the methods of recov-

ering the organic and inorganic values left in Steffen or other saccharate waste water or distillery slop as being the most economically feasible. It is upon this basis, therefore, that the present investigation is being conducted. The various assumptions on which the following patent survey is based may be stated as follows: (1) That the greatest need is for some method of utilization of the discard molasses and waste water of the beet-sugar industry; (2) that the maximum amount of molasses has been treated by the Steffen or other saccharate process to recover the maximum amount of sugar; (3) that the recovery of the organic and inorganic values in the waste water presents a hopeful outlook; and (4) that since methods of utilization have proven successful in other beet-sugar countries, such as Germany, some similar or analogous process might well be developed as an aid to the beet-sugar industry in the United States.

(f) COMPOUNDS FOUND IN WASTE MOLASSES.—Through the work of various investigators, chiefly in France and Germany, the nature of the various compounds to be found in beet-sugar waste waters has been made known. Among the possible products from beet molasses, exclusive of the sugar which may be almost entirely removed by means of saccharate processes, are the following: Methylamines, ammonia or ammonium sulphate, sodium or potassium cyanides, nitrogen bodies, such as betaine and glutaminic acid, alkali salts (K_2CO_3 , Na_2CO_3 , K_2SO_4), vegetable carbon and tar, and various minor products, such as methyl alcohol, glycerin, organic acids, and esters. These latter have received considerable attention in the patent literature because of their production from distillery slop or vinasses. In arranging the results of our patent search, the patents have been grouped according to the chief product of the process described. These groups, in most cases, have been subdivided into smaller groups of a closely related nature, in order that the development of any one phase of the art might be more readily followed. No importance should be attached to this arrangement beyond affording a key by which to classify the patents. A chronological scheme of listing has been used throughout, except in such cases where the work of one patentee over a short period can be more easily observed by grouping together.

In addition to those patents bearing directly on the subject of the patent group, others have been included which seem to offer suggestive chemical information as to properties and reactions, as well as methods of preparation from similar sources, as, for

instance, the obtaining of various by-products from peat, animal waste, and other industrial wastes aside from those of the beet-sugar industry.

II. PATENT OUTLINE AND SUMMARY.

GROUP 1.—AMINES.—Processes for producing methylamines from waste water. The patents indicate that little attention has been given to the preparation of amines as such from waste water or molasses. Methods for the preparation from other sources and the separation of the three methylamines are of interest from the research point of view.

GROUP 2.—AMMONIA.—(a) Production of ammonia directly from waste water, discard molasses, or other sugar wastes. (b) Production of ammonia from other industrial wastes. (c) Separation of the ammonia from processes of distillation or incineration. Means of absorbing, fixing, or purifying ammonia are so generally known in connection with the by-product coal industry that it was deemed inadvisable to make any attempt to cover this angle of the process completely. (d) Further processes for the production of ammonium salts from the constituents of the gases of dry distillation.

The principles used in the production of ammonia from waste water or molasses are included in a few well-defined methods: (1) Heating the waste water with an excess of alkali in any suitable container, said alkali sometimes being obtained from the waste-water ash. (2) Passing the gases from the dry distillation of waste water in contact with heated basic materials, such as lime, clays, bauxite, aluminates, or mixtures of waste-water ash and aluminates, which are subsequently treated for the recovery of the alkalies, alumina, etc. Various methods of using briquetted aluminates as a contact mass are described. (3) Calcining the waste water in contact with coke and steam to increase the yield of ammonia. (4) Ammonium salts are obtained directly by heating with acids, either with or without treatment with ferments.

Methods for obtaining ammonia from other industrial wastes are very similar to those noted above.

Under special conditions it is possible to prepare ammonium carbonate from the ammonia and carbon dioxide produced in the gases from the dry distillation of waste water.

GROUP 3.—APPARATUS.—Types of ovens, retorts, and miscellaneous equipment for all groups are described. In many cases

the descriptions of the process and the apparatus used are inseparable, and these have been introduced under their proper group classifications. Inasmuch as it is practically impossible to describe adequately a given type of apparatus without a diagram, illustrations of certain typical forms are introduced to simplify the description. The various types of apparatus are subdivided as follows: (a) Apparatus for incinerating and calcining waste water; (b) apparatus for evaporating and concentrating waste water; (c) apparatus for recovering ammonia from waste waters; and (d) apparatus for recovery of potash.

The development of retorts for dry distillation has been in the general direction of either rotating drums with stationary scrapers, or stationary retorts containing screw conveyors or other means of agitating and moving the incinerating mass. Many suggested pieces of equipment are highly complicated and could not be adequately reproduced, aside from the consideration of their practical utility.

GROUP 4.—CYANIDES.—In this group it was found necessary to limit the number of related patents to a small fraction of those actually gathered from the patent literature. The chemical principles entering into the production of cyanides by various high temperature and catalytic methods are so closely connected with those involved in their production from waste water that it proved difficult to limit the number included in the following lists. In general, however, where a patent was taken out on a related subject and it was known that the patentee was active in the field of our interest, such a patent was considered to have sufficient connection to warrant its inclusion.

The production of cyanides has been arranged in the following subgroups: (a) Production of cyanides from waste water direct; (b) production of cyanides from amines by special processes; (c) production of cyanides from various gas mixtures; (d) recovery of hydrocyanic acid from gas mixtures, and (e) preparation of cyanides in marketable form.

The present methods for the preparation of cyanides from waste water are developments of the original process of Ortlieb and Mueller ¹ in the use of some type of superheater through which the gases from the dry distillation are passed either directly or after the separation of the cyanizable and noncyanizable constituents. Reichardt and Bueb patented the first application of this principle, using a checkerwork of fire brick heated to a red

¹ Ger. 9409, Aktien Gesellschaft Croix (subgroup b).

or white heat as a cyanizing chamber. Subsequent patents have changed the form of the superheater to tubes made of quartz, zirconia, and other acidic materials.

One or all of the amines are also used in other ways than that described by Ortlieb and Mueller for producing cyanides, as in the process for passing the vaporized amine into molten sodium to produce the alkali cyanide directly.

By the use of catalysts, certain of the gases produced in the dry distillation of waste water may be made to combine to form hydrocyanic acid.

GROUP 5.—NITROGENOUS NONSUGARS.—Patents not previously listed under amines, ammonia, or cyanides, applying to nitrogenous compounds, such as betaine and glutaminic acid, are collected under the following groups: (a) Betaine and glutaminic acid from waste water and molasses, and (b) betaine prepared from other sources.

The methods for preparing betaine and glutaminic acid from waste water and molasses are, in general, variations of the treatment of those materials with either gaseous or aqueous hydrochloric acid and alcohol.

GROUP 6.—POTASH AND ALKALI SALTS.—Only those patents bearing directly on the waste-water problem have been taken for this survey. In the German files alone, there were innumerable related patents, dealing with alkali-salt systems with interesting chemical reactions and separations, which it was necessary to exclude.

The recovery of potash and alkali salts from the waste-water ash is accomplished by two general methods: (1) Evaporating, igniting, or calcining the waste water and leaching the resulting product in various ways to separate the potassium and sodium compounds. (2) Precipitation of the potash either with hydrofluosilicic acid or perchlorates.

Another form of utilization, developed to a considerable extent in America during the late war, is found in the direct use of the ash as a fertilizer.

GROUP 7.—RECOVERY OF MISCELLANEOUS PRODUCTS FROM WASTE WATER, MOLASSES, DISTILLERY SLOP, ETC.—This group includes all other products not previously mentioned, and is arranged under the following headings: (a) Glycerine, fatty acids, esters, etc.; (b) fertilizers, feed, etc.; (c) dyeing and mordanting materials; and (d) miscellaneous products—alkalies, phosphates, etc.

Glycerine and fatty acids are obtained from waste water by treatment of the dried residue with solvents, such as alcohol and acetone, or by fractional distillation under partial vacuum. The various specifications show slight changes from this procedure.

An interesting side-light on the use of waste water is as an agent in the dyeing and treating of wool, where the organic acids, particularly betaine acid, are the active reagents. In combination with ammonium molybdate, certain dyes may be obtained after treatment with sulphuric acid and alcohol which vary in color from light green to dark blue.

III. PATENT SYSTEMS OF VARIOUS COUNTRIES.

For those not familiar with the patent systems of the various countries the following information is given:

UNITED STATES.—Patents numbered consecutively from the beginning. Dates given: (1) Application, (2) issue or patenting.

GREAT BRITAIN.—Patents numbered consecutively in each year, a new series starting with each succeeding year until 1916, when a change was made to the system used in the United States. Previous to that time, therefore, it is necessary to have, in addition to the patent number, the year of its publication in order to properly identify the patent. Dates given: (1) Application, (2) complete acceptance.

FRANCE.—Numbering system similar to that of the United States. Previous to 1901 a description of the patent and one date only were published, while in many cases, at the discretion of the director of the French Patent Office, only the title of the patent, without any description or specifications, were published. These latter have been indicated "unpublished" in the following lists. Since 1901 complete dates and specifications are available. Dates given: (1) Application, (2) patenting, (3) publication.

GERMANY.—Numbering system similar to the United States. Dates given: (1) Patenting, (2) publication.

IV. TABULATION OF PATENTS.

1. GROUP I.—AMINES.

Ger. 5786. Aktien-Gesellschaft Croix. Pat. October 6, 1878; Pub. July 7, 1879.

Title: Process of manufacturing alkali carbonates using trimethylamine.

Describes the reaction between potassium or sodium chloride with trimethylamine bicarbonate to form the corresponding alkali carbonates. One part of potassium chloride is mixed with four parts of trimethylamine carbonate, and the mixture saturated with carbon dioxide to form potassium bicarbonate. Trimethylamine hydrochloride is left in solution.

Ger. 9376 (addition to Ger. 5786). Aktien-Gesellschaft Croix. Pat. September 9, 1879.

Specifies trimethylamine from beet molasses, as well as monomethylamine, dimethylamine, monopropylamine, monoisobutylamine in addition to trimethylamine.

Fr. 219180. Quantin. Pat. February 5, 1892.

Title: Separation of amines from ammonia by precipitation as magnesium ammonium phosphate.

To separate the methylamines from ammonia the crude mixture is distilled with milk of lime, and the distillate is collected in water, moist magnesium phosphate is then added, when, in the presence of much methylamine, the ammonia will be precipitated in 24 hours. The filtrate yields on distillation with milk of lime the methylamines free of ammonia.

Ger. 71328. Kossel and Schloemann. Pat. May 3, 1893; Pub. October 2, 1893.

Title: Separation of primary from secondary and tertiary amine bases.

Metaphosphoric acid is used to separate primary amine bases, diamines, and diimides from other bases. Amine bases and diamines of aromatic and aliphatic groups give compounds insoluble in water. The imid bases and nitrile bases give soluble metaphosphates.

Fr. 308407. Keppich. Pat. February 23, 1901.

Title: Process for separating amines from ammonical waters. (Unpublished.)

Ger. 270260. Schmitz, K. Pat. June 3, 1913; Pub. February 12, 1914.

Title: Process for the preparation of trimethylamine.

Trimethylamine is prepared from ammonium salts and formaldehyde by heating a mixture of dry ammonium salt and dry trioxymethylene or paraformaldehyde for a short time to a high temperature in open vessels provided preferably with a reflux condenser without the application of pressure.

Br. 14493/13. Bayer & Co. App. June 23, 1913; Acc. April 30, 1914.

Title: Improvements in and relating to the manufacture and production of dimethylaminomethane and dimethylamine.

Dimethylamine is prepared by treating trimethylamine salts with hypohalous acids and their salts, and treating the dimethylhalogen amine, such as dimethylchloroamine thus formed, after isolation, with reducing agents, such as bisulphite.

Br. 127740. Rintoul, Thomas, and Nobel's Explosives Co. App. June 15, 1918; Acc. June 12, 1919.

Title: Improvements relating to the preparation of amines.

Tertiary amines are separated from admixed primary or secondary amines by treating the mixture with ethyl chloroformate to convert the primary or secondary amines into urethans; the unattacked tertiary base is removed from the product by washing with dilute acid and is removed from the acid solution by treatment with alkali; the separated urethan is hydrolyzed to give the primary or secondary amine. An example is given of the treatment of crude dimethylaniline containing monomethylaniline.

Br. 128372. Rintoul, Thomas, and Nobel's Explosives Co. App. June 15, 1918; Acc. June 16, 1919.

Title: Improvements relating to the preparation of amines.

Tertiary amines are separated from admixed secondary amines by treating the mixture, at low temperatures, with phosgene to convert the secondary amine into the corresponding urea chloride; if the tertiary amine is in excess, the separation can be effected in one stage.

2. GROUP II.—AMMONIA.

(a) PRODUCTION OF AMMONIA FROM WASTE WATER, DISCARD MOLASSES, OR OTHER SUGAR WASTES.

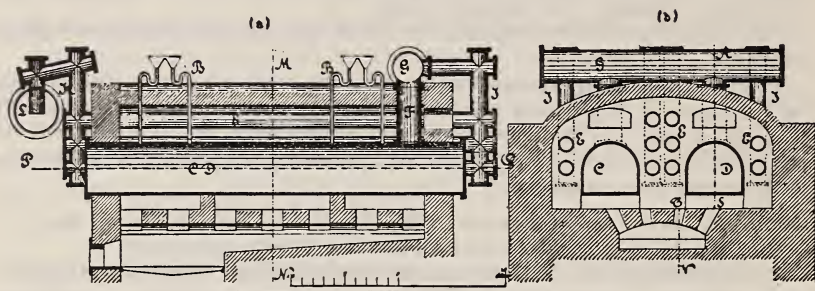


FIG. 1.—Ger. 15751.

Ger. 15751. Haring, Ehrenberg & Co., and Baswitz. Pat. April 2, 1880; Pub. December 6, 1881.

Title: Improved process for the dry distillation of molasses waste water, sugar elution water, and osmosis waste water for the recovery of ammonia.

Waste water is concentrated to 1.35 to 1.39 specific gravity and introduced into a retort at dark-red heat to cause immediate gasification. The gases are led through other heated tubes, at the side of the retort, which are filled with solid materials, such as clay, chalk, or chamotte. The chief product is ammonium sulphate and some methyl alcohol.

Ger. 11543. Ernst, E. Pat. February 4, 1880; Pub. October 16, 1880.

Title: Improvements in the process for conservation of nitrogen in waste water by the addition of sulphuric acid.

To concentrated waste water of 45–55° B. is added 15 per cent of 66° sulphuric acid while the waste water is hot; then 15 per cent dry powdered charcoal from soft woods is added and the whole changes to a dry mass without loss of nitrogen.

Ger. 13871. Ernst, E. Pat. October 6, 1880; Pub. May 30, 1881.

Title: Improvement in the dry distillation of molasses waste water.

Concentrated waste water is mixed with peat, calcium oxide, and fatty oil and the mixture dry distilled. The lime sets free ammonia and the oils enrich the fuel gases produced. Describes retort for process.

Ger. 14212 (addition to Ger. 11543). Ernst, E.

Title: Improvement in the process of utilizing molasses wastes through the addition of sulphuric acid and peat.

Specifies the use of all fluid molasses residues in the main patent.

Ger. 14433. Brosche, Sohn, F. X. Pat. July 9, 1880; Pub. July 28, 1881.

Title: Improvements in the process of recovery of nitrogen as ammonia from molasses waste, elution waste water, and osmosis waste water.

Ammonia is recovered from waste waters, as indicated in the title, by distillation over potash lime prepared from the waste-water ash and lime.

Ger. 15702. Badische Gesellschaft für Zucker Fabrikation. Pat. December 4, 1880; Pub. November 16, 1881.

Title: Improvements in the process of dry distillation of molasses waste water for the recovery of carbon, ammonia, methyl alcohol, and fuel gas.

Sodium hydroxide, prepared from the process itself, is added before distillation so that trimethylamine is not formed and ammonia only is recovered.

Ger. 43345. Meyer, E. Pat. August 28, 1887; Pub. April 28, 1888.

Br. 17347/87. Meyer, E. App. December 16, 1887; Acc. December 15, 1888.

Fr. 187573. Meyer, E. Pat. December 13, 1887.

U. S. 396705. Meyer, E., assignor to Carl Uhl & Co. App. September 15, 1888; Pat. Jan. 22, 1889.

Title: Obtaining ammonia and oxalic acid from sugar waste.

Waste water is added to caustic soda or potash in the proportions of 1 waste water to 8 caustic alkali, and heated. The greater part of the organic matter is destroyed, the nitrogen escaping as ammonia or amines, which are treated by well-known means, while in the molten mass a considerable quantity of oxalates are formed. The molten mass in the retort is run off and treated for oxalic acid, etc.

The patent specifies the addition of molasses or waste water in small doses and at intervals to prevent a violent evolution of ammonia.

Ger. 47190 (addition to Ger. 43345). Carl Uhl & Co. Pat. September 7, 1888; Pub. April 8, 1889. (See last paragraph above.)

U. S. 486647. Sternberg, L., assignor to American Sugar Refining Co. App. January 5, 1892; Pat. November 22, 1892.

Fr. 226024. Sternberg, L., assignor to American Sugar Refining Co. Pat. November 30, 1892.

Ger. 71408. Sternberg, L., assignor to American Sugar Refining Co. Pat. December 1, 1892; Pub. October 2, 1893.

Ger. 78442. Sternberg, L., assignor to American Sugar Refining Co. Pat. November 22, 1892; Pub. November 10, 1894.

Title: Process for obtaining ammonia and other salts from molasses.

Waste lyes from sugar manufacture are concentrated, then mixed with crushed coke, waste-water ash, etc., previous to calcining in closed retorts similar to those used for revivifying bone black. Superheated steam is introduced, and the nitrogen of the organic bodies is theoretically converted to ammonia. The ammonia liberated is led into sulphuric acid or into a tower of loose bricks, over which trickles a stream of ammonium sulphate solution containing free sulphuric acid. After absorption of the ammonia, the inert gases are passed back into the retort to prevent the decomposition of the ammonia. The residue in the retort is leached for alkalies or used to absorb more concentrated waste water.

Ger. 81401. Reichardt, H. Pat. February 12, 1893; Pub. November 5, 1895.

Title: Purification of ill-smelling gases from the waste-water ovens.

The alkaline waste water is carbonated by bringing it in contact with the hot gases from the waste-water oven, which contain carbon dioxide and are at a temperature of 250–400° C. The waste water is thus saturated and is then evaporated and the ash ignited. The volatile nitrogen-containing bases (chiefly amines) and ammonia are also purified.

Ger. 81341. Meyer, E. Pat. August 28, 1894; Pub. May 4, 1895.

Title: Process for the dry distillation of molasses waste water.

The process yields ammonia, which is set free by mixing lime-free molasses residues with an excess of alkali aluminum phosphate, the ammonia being absorbed and purified in known manner. Treatment of the raw alkaline aluminum phosphate yields aluminum oxide, calcium phosphate, potassium and sodium phosphates or a composite fertilizer direct. Lime is removed from the molasses to prevent reversion of the phosphate.

Ger. 86400. Offene Handelsgesellschaft. H. & W. Pataky. Pat. July 1, 1894; Pub. March 20, 1896.

Title: Recovery of ammonia and alkali carbonates from the residual lyes of desugarizing processes and from the manufacture of alcohol.

The lyes are evaporated to 70° Brix and added to $\frac{1}{2}$ to 1 part of calcined alumina. This mixture is heated in iron retorts at red heat. The charge does not sinter. Ammonia is absorbed by water or sulphuric acid. The fuel gases are used for heating the retorts. The loose residue in the retorts is leached with water, the insoluble portions being reused until the alumina is exhausted. Potassium and sodium aluminates or carbonates and aluminum hydroxide are removed from the process, depending upon the method of treatment by evaporating or carbonating.

Ger. 89147. Matthiessen, F. O. Pat. February 21, 1895; Pub. October 7, 1896.

Title: Process of manufacture of ammonia from volatile organic nitrogenous substances, using aluminate contact masses.

Gases from the distillation of molasses waste water are conducted over glowing contact substances consisting, in the main, of alkali or alkaline earth aluminates, formed by heating bauxite and waste-water ash. Their reaction capacity is unlimited, as they do not combine with carbon dioxide and their basicity is constant.

Ger. 93397. Matthiessen, F. O. Pat. February 21, 1895; Pub. July 14, 1897.

Title: Process for obtaining ammonia from molasses waste water.

Molasses waste, alkali aluminate, and alumina are mixed and dried at a low temperature to keep the mass from sintering, then raising the temperature to liberate the ammonia. The alkali aluminate is a permanent basic contact body. After ignition, the contact mass is pulverized and used again, while any excess is treated as outlined in Ger. 86400. This process requires a lower temperature than previous patents. The contact body is formed into briquets.

U. S. 557166. Sternberg, L., assignor to American Sugar Refining Co. App. November 9, 1895; Pat. March 31, 1896.

Br. 7002/96. Sternberg, L., assignor to American Sugar Refining Co. App. March 31, 1896; Acc. August 15, 1896.

Title: Process of obtaining ammonia from waste-sugar lyes.

An aluminate of an alkali or alkaline earth is used as a carrier for waste lye or molasses. Upon being heated the gases are passed through potassium aluminate and alumina and changed to ammonia. The aluminate is made into bricks of $1\frac{1}{2}$ parts of bauxite to 1 part of crude potash. The lye is concentrated to 75° Brix, mixed with alumina and an aluminate to form a plastic mass, forming into bricks and then drying and heating to redness, forming an incandescent contact body which transforms nitrogen into ammonia.

Ger. 118795. Besemfelder, E. R. Pat. June 11, 1899; Pub. March 20, 1901.

Ger. 121222 (addition to Ger. 118795). Besemfelder, E. R. Pat. June 11, 1899; Pub. March 20, 1901.

U. S. 689780. Besemfelder, E. R. App. September 29, 1899; Pat. December 24, 1901.

Title: Method of making ammonia from waste products.

The process is carried out by one of the following methods: (1) Dry distills liquors, conducting the gas over heated aluminate; through alkali to absorb cyanides, then over calcium diphosphate. (2) Dry distills in contact with alumina, conducting the gases as in (1). (3) Distills as in (2), adding coke to the mixture, and while in a glowing state blasting with air and steam to form water gas, conducting same in contact with the quantity of the waste and alumina undergoing distillation and then conducting the gas as in (1).

Ger. 209114. Effront, J. Pat. December 11, 1906; Pub. April 20, 1909.

Br. 28108/06. Effront, J. App. December 10, 1906; Acc. May 30, 1907.

U. S. 868976. Effront, J. App. December 13, 1906; Pat. October 22, 1907.

Fr. 369630. Effront, J. App. August 22, 1906; Pat. November 20, 1906; Pub. January 16, 1907.

Title: Process for the utilization of nitrogen in distillery residues.

The residues are treated with sodium bisulphate and heated in a closed vessel to 180–200° C., a current of air meanwhile passing through the vessel. About one-half the ammonia is recovered in the form of salts. The residue is then powdered and heated to 700° C. in a current of superheated air and steam. This distillation contains the remainder of the ammonia and the methyl alcohol. Molasses residues are treated preferably with resin in place of the bisulphate, and dried in an atmosphere consisting alternately of air and carbon dioxide, the vapors being collected in acid.

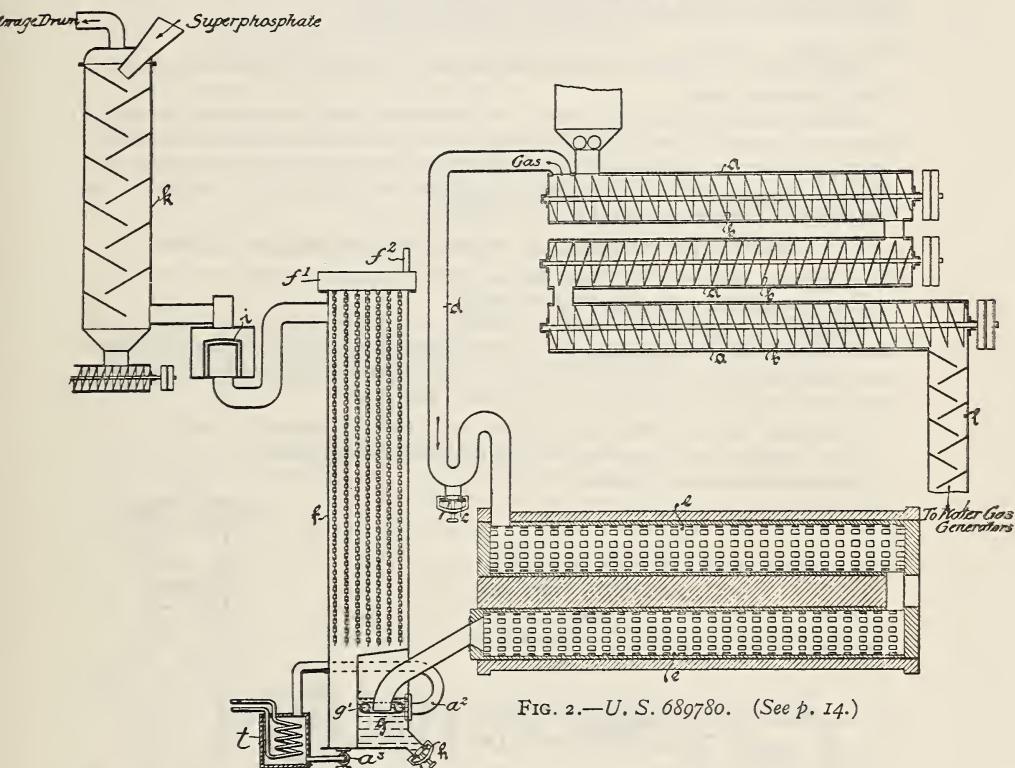


FIG. 2.—U. S. 689780. (See p. 14.)

Fr. 382689. Effront, J. App. October 8, 1907; Pat. December 14, 1907; Pub. Feb. 13, 1908.

Title: Process of recovery of ammonia by fermentation of nitrogenous organic substances.

The residues are fermented with a butyric ferment in an alkaline medium. The ferment is acclimatized in the presence of acid amides.

Fr. Addition No. 9148. Effront, J. App. April 1, 1908; Pat. June 17, 1908; Pub. August 29, 1908. (See Fr. 382689.)

Ger. 215531. Societe Anonyme de Recuperation de Produits Chimiques "Procede Effront." Pat. May 8, 1908; Pub. October 27, 1909.

Title: Process of obtaining ammonia and fatty acids through the action of ferments of albumen and their reaction products.

Distillery wash, sugar manufacture waste, and peat extracts may be treated with ferments under certain conditions for the formation of ammonia and fatty acids (acetic, propionic, butyric).

Ger. 270325. Gouthiere, H. and Cie., and Ducancel, P. Pat. November 7, 1911; Pub. February 16, 1914.

Fr. 442923. Gouthiere, H. and Cie., and Ducancel, P. App. July 1, 1911; Pat. July 2, 1912; Pub. September 12, 1912.

Title: Process for the preparation of ammonium sulphate.

A mixture of vinasse with ash from a previous distillation is dry distilled in the presence of water vapor and the ammonia is collected in sulphuric acid. The fuel gases produced are used for heating the retorts. An addition patent, dated October 18, 1912, covers the obtaining of glycerine from the process.

Ger. 339302. Muhlert, F. Pat. December 28, 1917; Pub. July 21, 1921.

Title: Process of obtaining ammonium salts and cyanogen compounds by gasification of nitrogen wastes.

The raw distillation gases, or better, the condensate which is produced by cooling the gases, is distilled, and the mixture of gases of ammonia and organic bases is led into appropriate acid of such concentration as to just neutralize the ammonia, etc. The water vapor of the distillate is separated from the gases in known manner. The nonabsorbed gases are cyanized in known manner.

(b) PRODUCTION OF AMMONIA FROM OTHER INDUSTRIAL WASTES.

U. S. 216323. Grouven, H. App. May 9, 1878; Pat. June 10, 1879.

Title: Manufacture of sulphate of ammonia.

Ammonium sulphate from turf and similar material is obtained by mixing with chalk, driving off the vapors and gases by heat, and decomposing the vapors by a contact mass; converting the ammonium carbonate to ammonium sulphate in the presence of calcium sulphate; purifying the ammonium sulphate.

U. S. 247046. Grouven, H. App. June 6, 1881; Pat. September 13, 1881.

Title: Process of and apparatus for making ammonium sulphate.

Apparatus for the above patent.

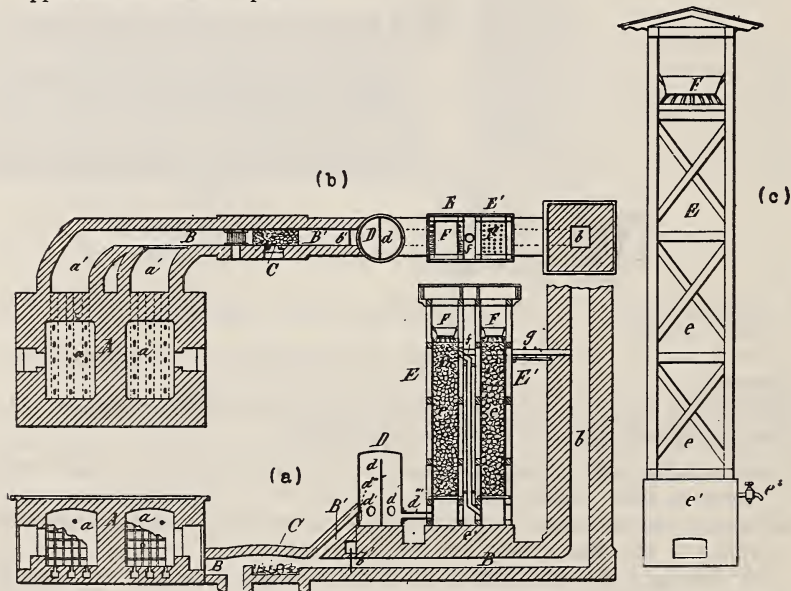


FIG. 3.—U. S. 232991.

U. S. 232991. Lorenzen, H. P. App. January 29, 1880; Pat. October 5, 1880.

Title: Method and apparatus for obtaining ammonia.

Ammonia is recovered from the distillation of nitrogenous substances by passing the gases through incandescent lime, cooling, and absorbing in sulphuric acid.

U. S. 278823. Rickman and Thompson. App. October 7, 1882; Pat. June 5, 1883.
Title: Manufacture of ammonia and its salts.

Ammonia and its salts are prepared from animal excreta and other nitrogenous material by fermentation and distillation.

U. S. 282411. Terne, B., assignor of three-fourths to D. and E. P. Baugh, Philadelphia, Pa. App. December 11, 1882; Pat. July 31, 1883.

Title: Process of treating tank waters of slaughter houses.

Partial dry distillation of concentrated tank waters, leaving portion of the ammonia-containing materials in the residue to be used as a fertilizer.

U. S. 342237. Van Ruymbeke, J., assignor to National Chemical & Fertilizer Co.

App. August 4, 1885; Pat. May 18, 1886.

Title: Process of obtaining ammonia.

By showering the nitrogenous liquors through forced currents of air within closed vessels over porous substances charged with putrid ferments.

U. S. 342238. Van Ruymbeke, J., assignor to National Chemical & Fertilizer Co.

App. July 6, 1885; Pat. May 18, 1886.

Title: Process of making a fertilizer from tank waters.

By heating the tank waters to dryness at about 460° F.

U. S. 351412. Van Ruymbeke, J., assignor of one-half to W. F. Jobbins. App. July 27, 1885; Pat. October 26, 1886.

Title: Process of obtaining ammonia and illuminating gas from tank waters.

By dry distillation at not over 500° F.

U. S. 374618. Nast, W. F. App. May 4, 1887; Pat. December 13, 1887.

Title: Obtaining ammonia from manure, etc.

By heating the manure, etc., and conducting the gases in contact with heated basic materials, such as milk of lime, containing admixed sodium chloride (2 per cent) to facilitate or complete the reaction.

U. S. 488207. Kuntze, P. App. December 31, 1891; Pat. December 20, 1892.

Ger. 62589. Kuntze, P. Pat. April 8, 1891; Pub. December 27, 1892.

Title: Process of and apparatus for making ammonia.

Nitrogen in organic matter is left behind if the temperature is not carried high enough. Some escapes in the form of nitrites, organic bases of pyrrol and pyridine, amines, etc. The quantity of nitrogen products increases with progress of the dry distillation. After driving off the tar, the temperature rises and nitrogen as ammonium cyanide is formed in considerable quantity. This process tends to fix all the nitrogen as ammonia by calcining and heating. Describes a complicated apparatus for obtaining ammonia from peat.

Ger. 87061. Pieper, C. (Inc.). Pat. June 28, 1894; Pub. May 20, 1896.

Title: Process of treating nitrogenous substances for the recovery of ammonia and coke.

Peat or nitrogenous bodies are heated in retorts heated from the outside, from which the air is excluded. Sufficient steam is introduced for ammonia formation, also diminishing the loss of btu's if water gas forms. The temperature of the reaction is kept under the dissociation temperature of ammonia. The mixture of ammonia, hydrogen, and carbon dioxide passes through sulphuric acid. Nitrogen-free carbon remains in the retort. It appears that the evolution of gas from the peat, etc., must be complete before the steam is admitted.

Ger. 93705. Pieper, C. (Inc.), and Fellner and Ziegler. Pat. November 10, 1896; Pub. September 11, 1897.

Title: Process of recovery of ammonia from nitrogenous substances in continuous operation.

Improvement in Ger. 87061 to permit continuous operation. The material is conducted through a series of steaming chambers, which are so constructed with their fuel-gas supply, etc., that one or more of the units may be cut out without interfering with the process. The products of the gasification are kept separate. Steam used is recovered from the drying process.

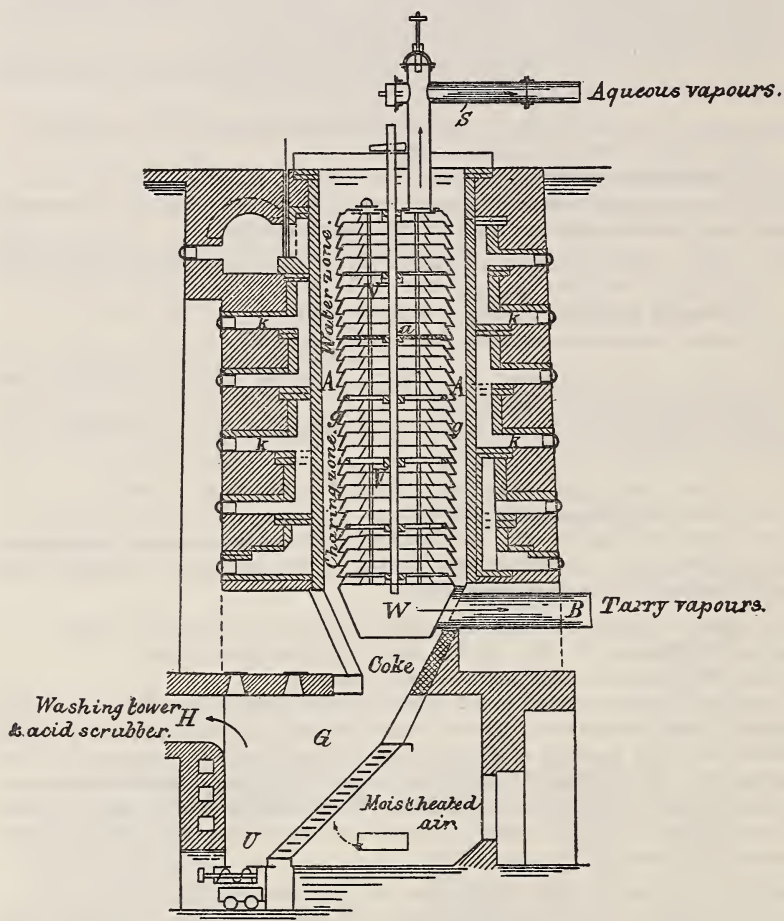


FIG. 4.—U. S. 488207. (See p. 17.)

Ger. 93706. Pieper, C. (Inc.), and Fellner and Ziegler. Pat. November 10, 1896; Pub. August 31, 1897.

Title: Process of utilization of nitrogenous substances.

Gases from the first distillation and steaming process are kept separate. The distillation products are freed from dust and dried, all condensable products removed, while the tar-free gaseous products of the steaming process are washed to remove the dust, and are then passed through acid to remove the ammonia.

Ger. 93746. Pieper, C. (Inc.). Pat. December 17, 1896; Pub. September 11, 1897.

Title: Process for the recovery of ammonia from nitrogenous substances and coke as per patent 87061.

A change in Ger. 87061. The distillation raw product is treated in a special apparatus with superheated steam in such quantity as is necessary for the decomposition of the material. The steam then meets only nitrogenous coke. Recovery of the ammonia is greater, and the time required is shorter. Makes use of countercurrent of coke and steam.

Fr. 353970. Guerrero, W. de Smirnoff. App. May 4, 1905; Pat. July 12, 1905; Pub. September 25, 1905.

Title: Process and apparatus for obtaining ammonium salts and other bodies.

Ammonium salts, methylene, acetates, tar, and charcoal are obtained from the dry distillation of residues from olives.

(c) SEPARATION OF THE AMMONIA FROM PROCESSES OF DISTILLATION OR INCINERATION.

Ger. 23594. Steffens, H. Pat. January 13, 1883; Pub. August 18, 1883.

Title: Recovery of ammonia from alcohol of desugarizing plants by precipitation with sulphuric acid.

Ger. 24549. Steffens, H. Pat. April 5, 1883; Pub. October 3, 1883.

Title: Recovery of ammonia from alcohol of desugarizing plants by precipitation with sulphuric acid or acid salts.

As well as sulphuric acid for the precipitation of ammonia from alcohol, carbon dioxide, phosphoric acid, potassium acid sulphate, sodium acid sulphate, or potassium acid phosphate may be used.

Fr. 331077. Societe F. Brunck. App. April 9, 1903; Pat. July 17, 1903; Pub. August 31, 1903.

Fr. Addition No. 3955 to above. Societe F. Brunck. App. November 5, 1904; Pat. February 2, 1905; Pub. April 5, 1905.

Title: Improvements in extracting ammonia from the gaseous products of dry distillation.

The gases are washed with concentrated sulphuric acid at 80–85° C. and the ammonium sulphate recovered directly in solid form. The addition patent specifies certain changes in the apparatus.

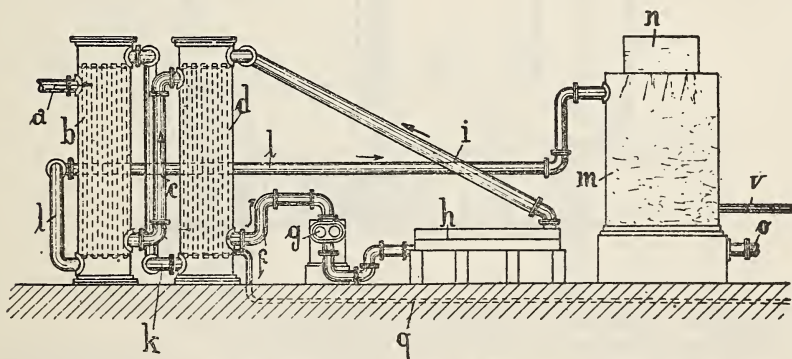


FIG. 5.—U. S. 846035. (See p. 20.)

U. S. 830983. Feld, W. App. January 4, 1906; Pat. September 11, 1906.

Title: Process of treating coal gas for extracting tar, water, and ammonia.

Tar and water are separated by means of sprays at a temperature between 40 and 500° C., preferably between 100 to 200° for tar, and above 40° and under 120° for water. Water is separated without absorbing ammonia, which is then recovered.

U. S. 851349. Feld, W. App. January 18, 1907; Pat. April 23, 1907.

Title: Extracting ammonia from gases.

Air is introduced into the retorts. The tar and water are separated in the usual way, and the gas passed into gypsum held in solution and suspension. Gases are produced from materials which are such that the gases contain less than 1 molecular proportion of carbon dioxide to 2 molecular proportions of ammonia. All ammonia is absorbed as above.

U. S. 846035. Koppers, H. App. September 2, 1905; Pat. March 5, 1907.

Title: Ammonia recovery apparatus and process.

The gas is cooled below 72° , the tar condensed and separated, the gas washed with acid to make a liquor which is saturated with the waste vapors obtained by treating said condensate with lime and steam.

U. S. 953959. Koppers, H. App. January 15, 1908; Pat. April 5, 1910.

Title: Recovering ammonia and tar from distillation gases.

By cooling to condense tar and water, separating the tar, scrubbing the gas with water to form an aqueous solution of ammonia. The condensate and aqueous solution are passed through a distilling column to separate ammonia, mixing the vapors from the distilling column with preheated air and conducting the mixture to a saturation bath containing sulphuric acid.

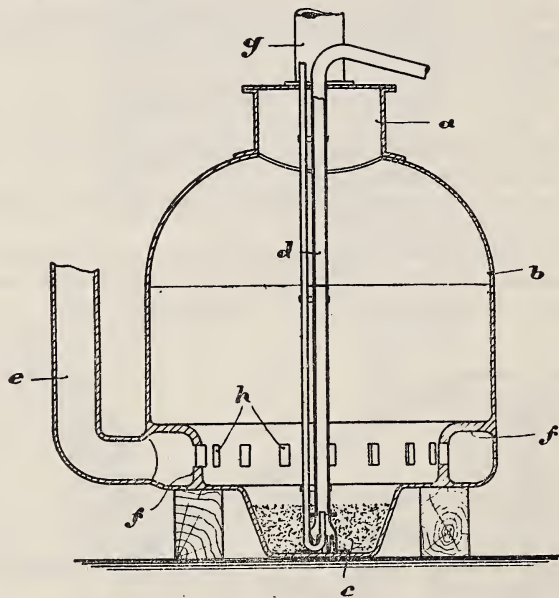


FIG. 6.—U. S. 953960.

U. S. 953960. Koppers, H. App. May 20, 1909; Pat. April 5, 1910.

Title: Recovering ammonia and tar from distillation gases.

Describes an ammonia saturation vessel for carrying out the process described in U. S. 953959.

U. S. 952560. Caro, N. App. June 28, 1909; Pat. March 22, 1910.

Title: Process of obtaining ammonium sulphate from gases.

By cooling the gases to $15-20^{\circ}$ to separate tar and ammonia water, distilling the latter, partially drying the vapors thus formed, mixing them with the previously cooled distillation gases and conducting the mixture into a solution of ammonium sulphate of $29-35^{\circ}$ B. containing about 5 per cent free sulphuric acid.

(d) FURTHER PROCESSES FOR THE PRODUCTION OF AMMONIUM SALTS FROM THE GASEOUS CONSTITUENTS OF DRY DISTILLATION.

Ger. 237524. Bueb, J., and Deutsche Continental Gas Gesellschaft. Pat. August 3, 1909; Pub. August 22, 1911.

Ger. 246017. Bueb, J., and Deutsche Continental Gas Gesellschaft. Pat. April 29, 1910; Pub. April 22, 1911.

U. S. 1018406. Bueb, J. App. April 12, 1910; Pat. February 27, 1912.

Title: Manufacture of ammonium carbonate.

Ammonia, carbon dioxide, and steam are passed through a tube heated above the decomposition point of ammonium carbonate and then cooled below 60° in a final chamber. The heat necessary for the reaction may be obtained by passing the tube through water heated to 60–90° C. The salt is deposited as a crust on plates cooled by water. The percentage of ammonia can be regulated by the temperature of the water through which the tube passes before reaction.

Ger. 252276. Bueb, J., and Deutsche Continental Gas Gesellschaft. Pat. September 12, 1909; Pub. October 22, 1912.

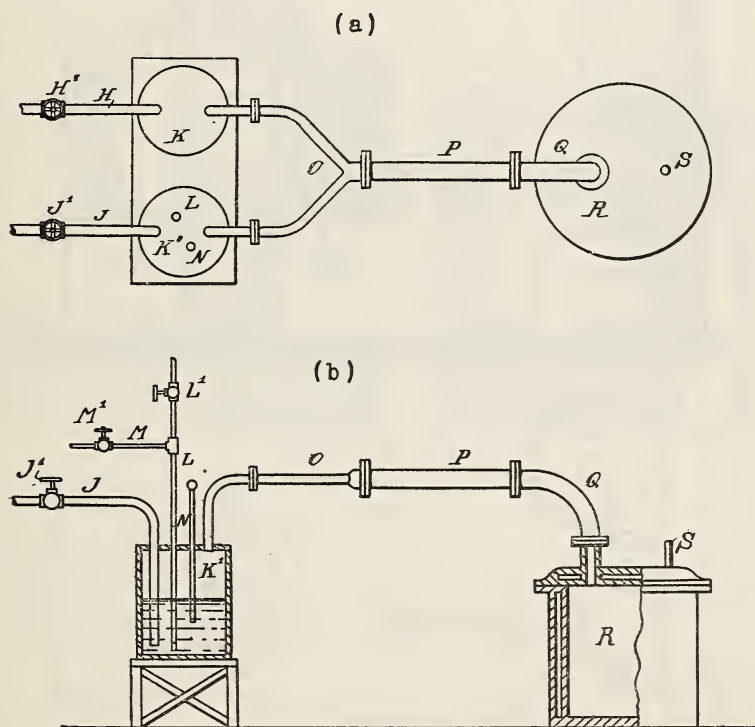


FIG. 7.—U. S. 1004361.

U. S. 1004361. Bueb, J. App. September 21, 1910; Pat. September 26, 1911.

Title: Method of making ammonium carbonate.

By adjustment of the temperature of the water used for heating the gases prior to the reaction a product may be obtained which contains any desired percentage of ammonia between 20 and 40 per cent. Special apparatus for the process is described.

CROSS REFERENCES.

Manufacture of Ammonia:

- Ger. 9409. Aktien-Gesellschaft Croix. Group 4(b).
 Fr. 485597. Dupont, L. Group 7(d).
 Ger. 232878. Hauff. Group 4(c).
 U. S. 1145484. Huber and Poindexter. Group 4(a).
 Fr. 385139. Riviere, L. Group 6.
 Ital. 135224. Vigano and Soldi. Group 6.

3. GROUP III.—APPARATUS.

(a) APPARATUS FOR INCINERATING AND CALCINING WASTE WATER, ETC.

Fr. 75927. Wagner. Pat. April 30, 1867.

Title: Double effect reverberatory furnace for the incineration of distillery vinasse and of all combustible material. (Unpublished.)

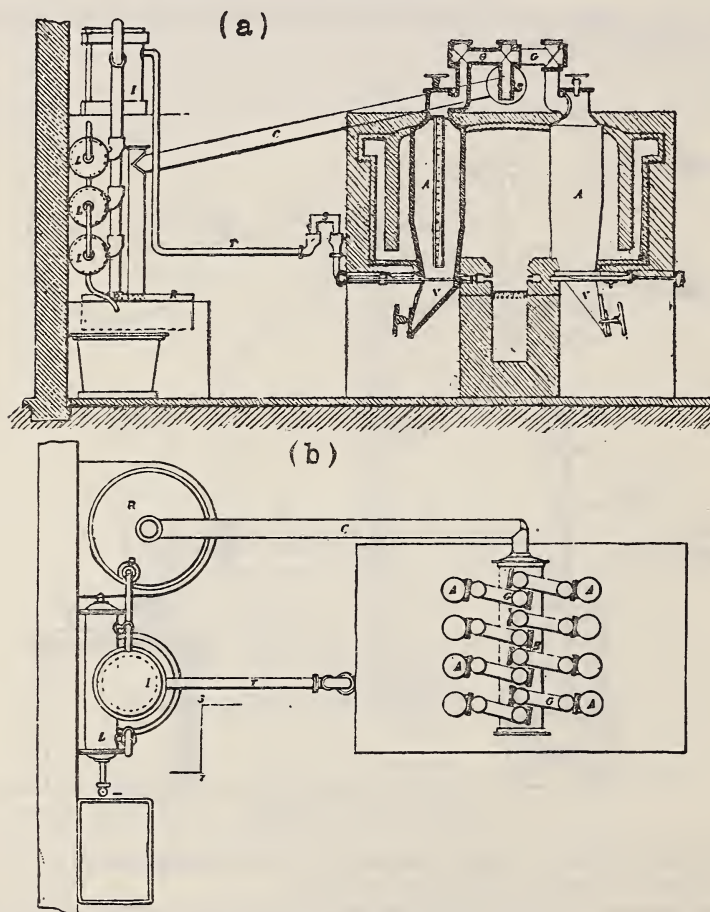


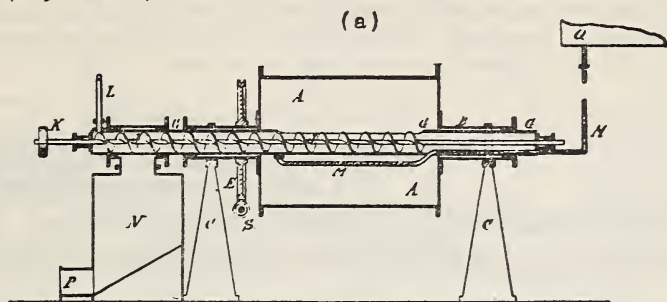
FIG. 8.—Fr. 122938.

Fr. 122938. Houzeau and Goutierre. Pat. March 1, 1878.

Title: Economic furnace for calcination and distillation of all materials with recovery of utilizable volatile products. (Unpublished.)

Fr. 139529. Farinaux and Laine. Pat. November 8, 1880.

Title: Furnace for evaporating and incinerating wool washings, vinasses, and such lyes. (Unpublished.)



(c)

(b)

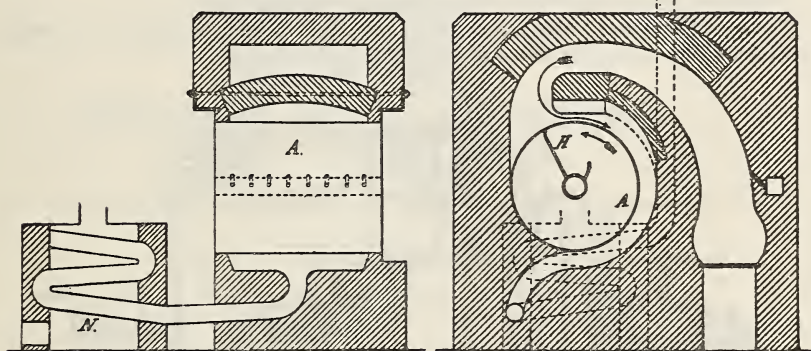


FIG. 9.—Ger. 17874.

Ger. 17874. Lederer and Gintl. Pat. May 28, 1881; Pub. April 24, 1882.

Title: Apparatus for the continuous carbonizing and distillation of molasses waste water.

Consists of a slowly revolving drum, mounted on a fire-brick setting through which gases are burned, into which molasses residues are pumped. As the dried residues reach the top they are scraped off and fall onto a conveyor screw and are taken outside the drum. The gases are taken off at another outlet.

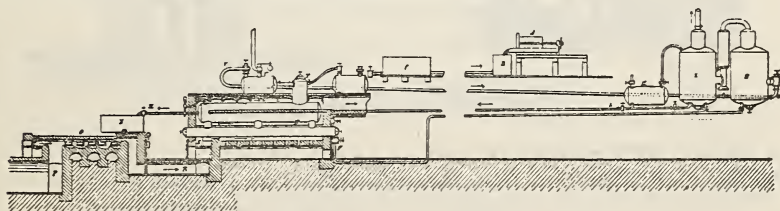


FIG. 10.—Fr. 154291.

Fr. 154291. Gamer, L. Pat. March 14, 1883.

Ger. 26236. Gamer, L. Pat. June 3, 1883; Pub. May 22, 1884.

Title: Continuous carbonizing retort for waste water with inclined separating walls on the hearth bed and dumping pit.

A set of ovens, each 4 m long and 1.6 m wide are arranged in a single setting. Several gates give access to the retorts. In each retort is an inclined fire brick which separates the floor of the oven into sections. Concentrated waste water enters the back of the oven and is pushed over these bridges toward the front section, where the incineration is completed. A grating is so arranged as to start the incineration process, but the oven is closed tight after being once started.

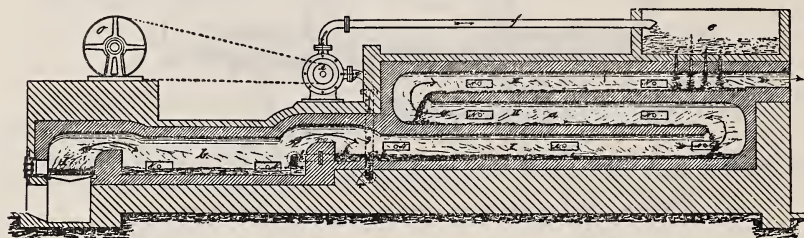


FIG. 11.—Ger. 26420.

Ger. 26420. Proeber, G. Pat. May 20, 1883; Pub. April 4, 1884.

Title: Oven for the evaporation and calcination of waste water.

The oven is arranged in a set of steps, or stages, one over the other, over which the liquor is circulated by means of a pump.

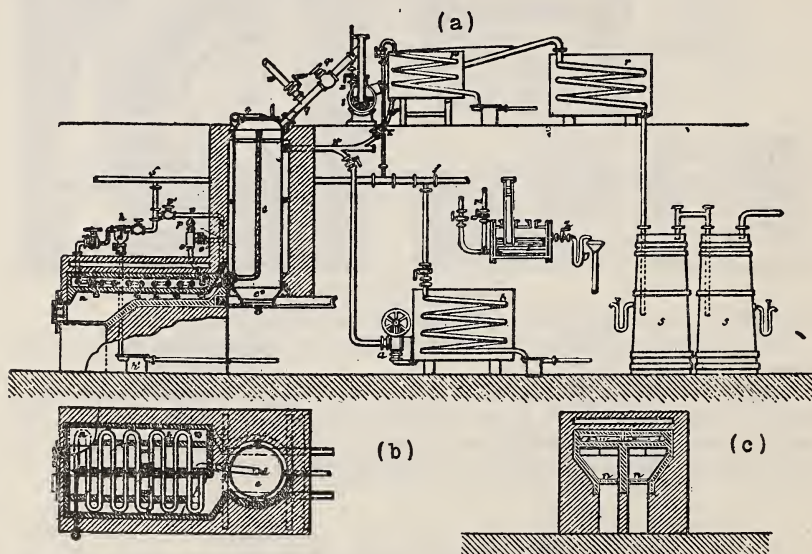


FIG. 12.—Fr. 184905.

Fr. 184905. Zwillingger, A. Pat. July 20, 1887.

Ger. 38419. Zwillingger, A. Pat. January 30, 1886; Pub. February 8, 1887.

Title: Improvement in the process of carbonizing molasses waste water.

An apparatus for combining the use of flue gases and superheated steam to evaporate waste water with the provision to absorb by-products.

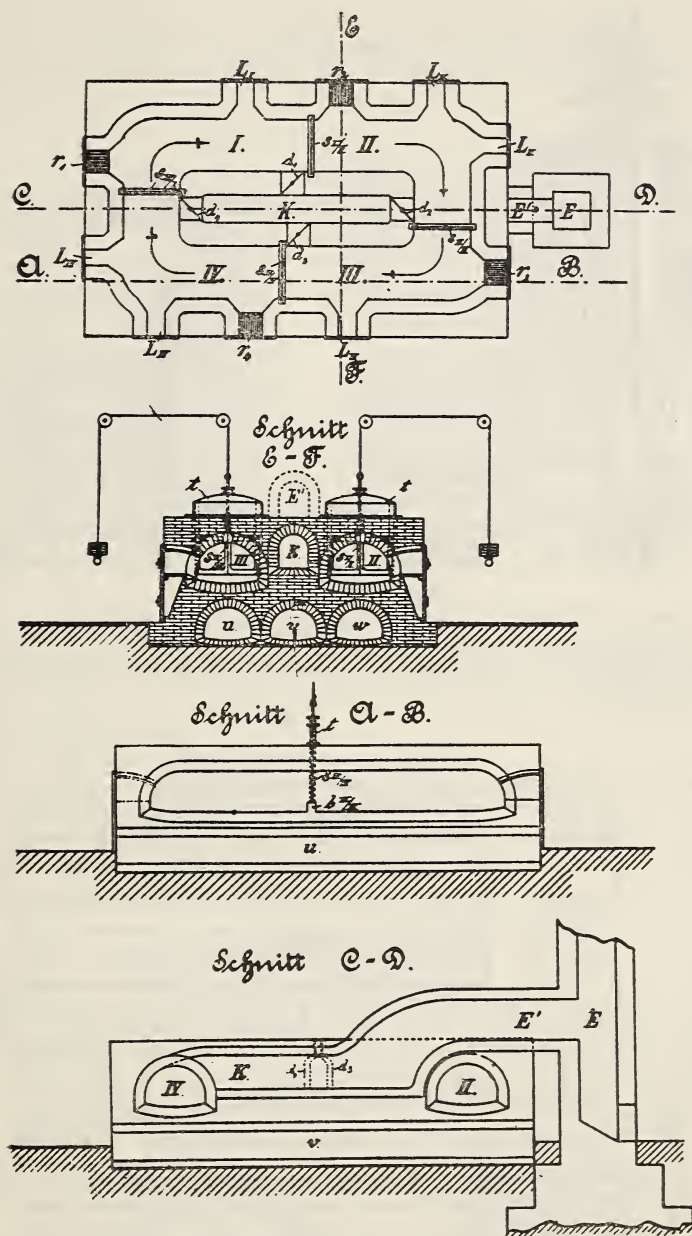


FIG. 13.—Ger. 61336.

Ger. 61336. Schwager, J. Pat. June 27, 1891; Pub. March 8, 1892.

Title: Furnace for the evaporation, distilling, and coking of organic substances contained in lyes.

A complicated and impractical series of ovens with many gates, valves, etc.

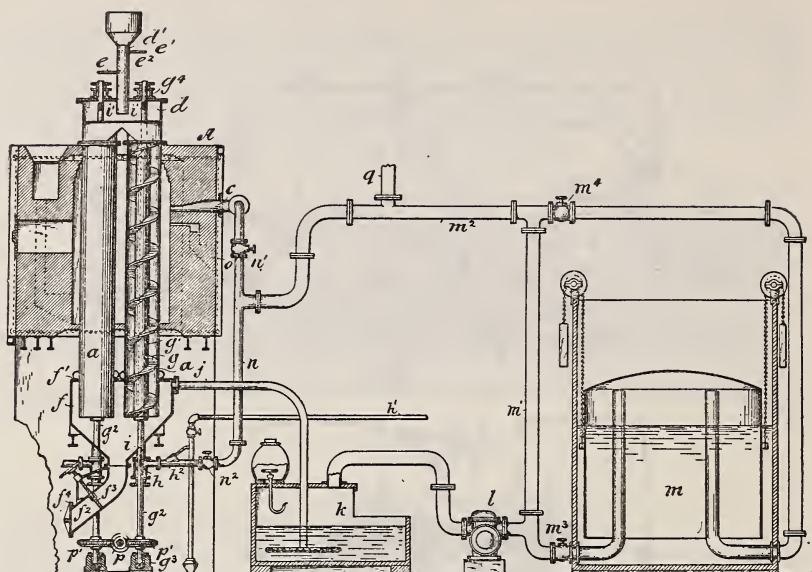


FIG. 14.—U. S. 522357.

U. S. 522357. Sternberg, L. App. October 19, 1893; Pat. July 3, 1894.

Title: Apparatus for obtaining ammonia.

Vertical retort for the distillation of nitrogenous liquids to produce ammonia or ammonium salts.

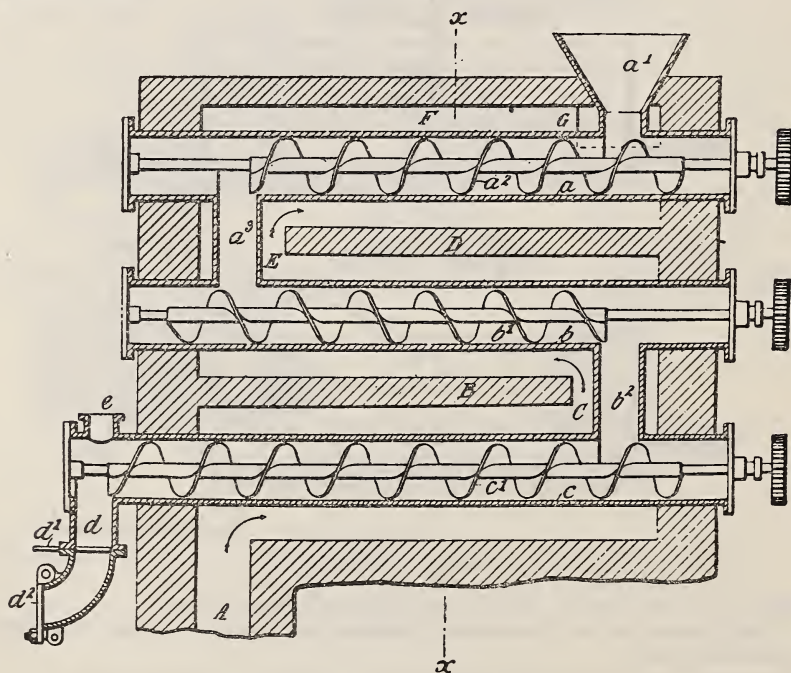


FIG. 15.—Ger. 105638. (See p. 27.)

Ger. 105638. Sternberg, L. Pat. August 10, 1897; Pub. September 22, 1899.

Title: Process of obtaining ammonia from residues of desugarizing or distillery slop.

Three retorts, one above the other, each equipped with a conveying screw for moving the mass from one retort to the one below.

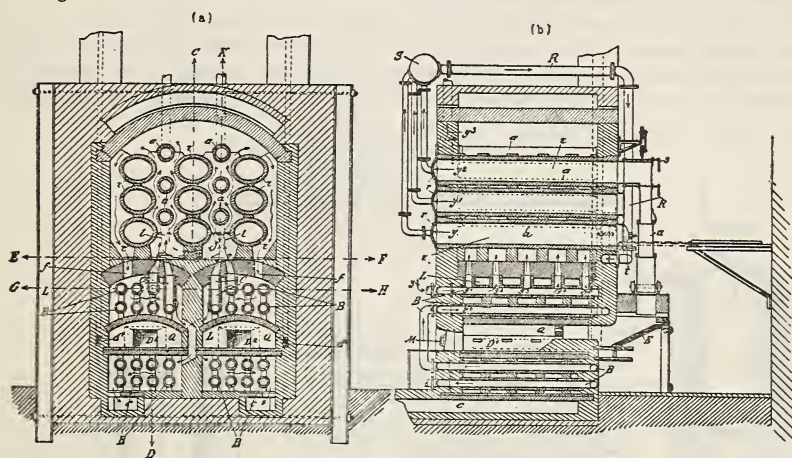


FIG. 16.—Ger. 108724.

Ger. 108724. Wenck. Pat. March 23, 1898; Pub. February 27, 1900.

Title: Combined distilling and calcining oven.

A set of horizontal ovens in a setting so arranged that the gases liberated are first freed of ammonia, returned through pipes between the retorts to convert further nitrogen to ammonia, and then conducted beneath the retorts to be used as fuel gas for heating the retorts.

Fr. 289951. Waissbein. Pat. June 15, 1899.

Title: Process of dry distillation or fractionation of solid or liquid matter by means of uncondensable gaseous products of the same distillation. (Unpublished.)

Austr. 1284. Pečekér Zuckerraffinerie. Pat. January 15, 1900; Pub. May 10, 1900.

Fr. 291245. Pečekér Zuckerraffinerie. Pat. July 28, 1899.

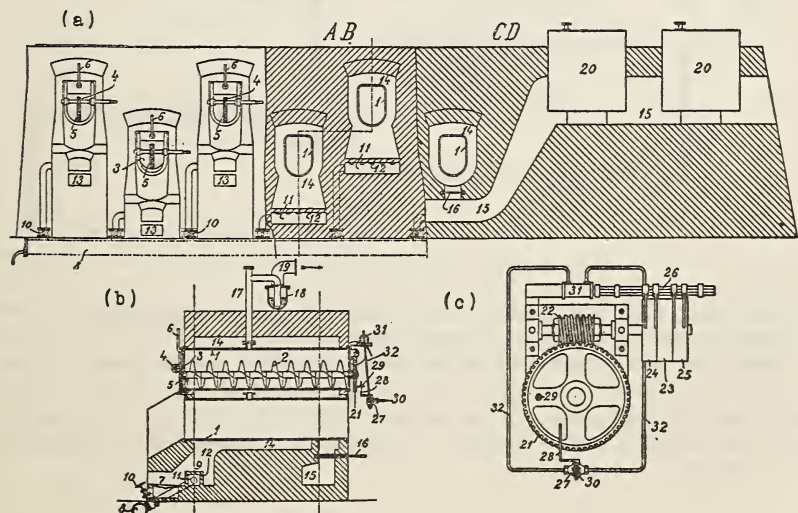


FIG. 17.—U. S. 666657. (See p. 28.)

U. S. 666657. Fischer, H. App. August 8, 1899; Pat. January 29, 1901.

Br. 15815/99. Fischer, H. App. August 2, 1899; Acc. October 28, 1899.

Title: A process of, and apparatus for, obtaining ammonia and its salts from spent liquor in sugar factories, and from vinasses.

The liquor is evaporated in special pans built into the flues. Each retort has its own heating system. The evaporated liquor is fed into troughs in which are screws for agitating and conveying. The gases are conducted through a tar condenser, ammonia scrubber, and to a gasometer from whence they are burned under the retorts.

Fr. 296955. Sudre and Thierry. Pat. February 7, 1900.

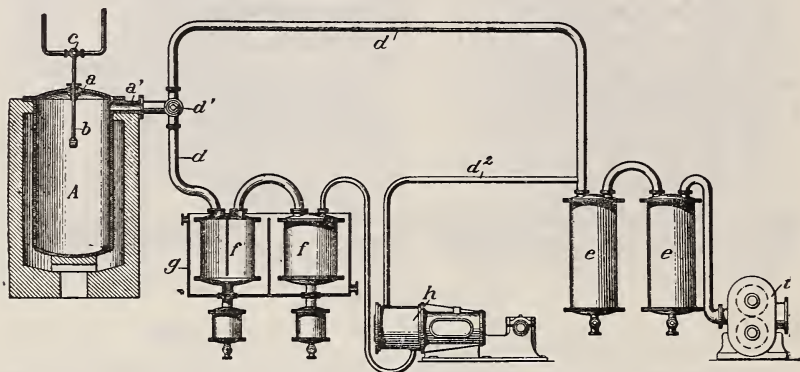


FIG. 18.—U. S. 669812.

U. S. 669812. Sudre and Thierry. App. February 15, 1900; Pat. March 12, 1901.

U. S. 712343. Sudre and Thierry. App. February 18, 1901; Pat. October 28, 1902.

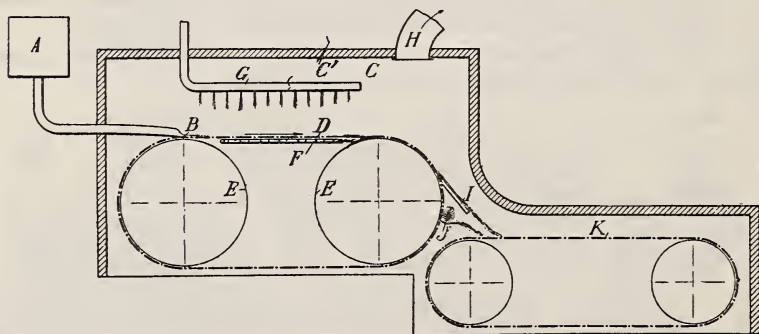


FIG. 19.—U. S. 732234.

U. S. 732234. Sudre and Thierry. App. August 29, 1900; Pat. June 30, 1903.

Title: Process and apparatus for treating distiller's wash.

Causticized wash is heated up to 500° C. to recover all possible products. The apparatus consists of a combustion chamber and absorption train. Improvements are indicated in the apparatus in the various patents to enable the patentee to recover glycerine in addition to other products. The apparatus is represented by complicated plan diagrams.

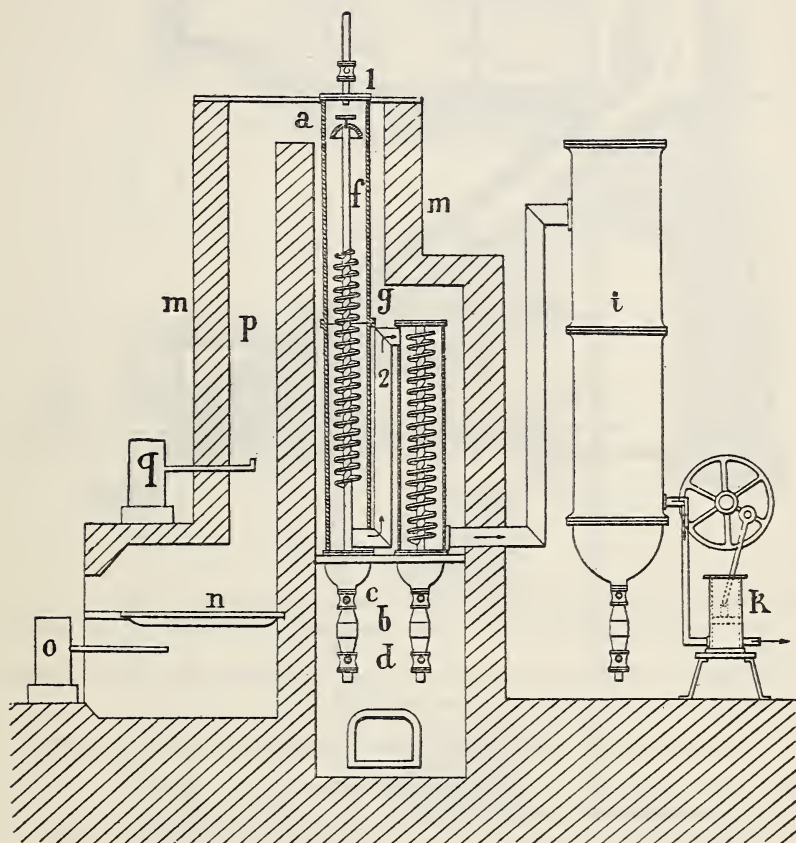


FIG. 20.—U. S. 810442.

U. S. 810442. Sudre, C. App. January 6, 1904; Pat. January 23, 1906.

Title: Process of treatment of vinasses.

A process for the extraction of glycerine in the form of a mixture of glycerine and water to be concentrated and producing a residue containing all the ammonia, which consists in rendering the vinasses acid and causing them to flow in a vacuum along a surface heated to a maximum temperature of 300°C . and causing the steam to have a gyratory movement to free it of the particles carried with it.

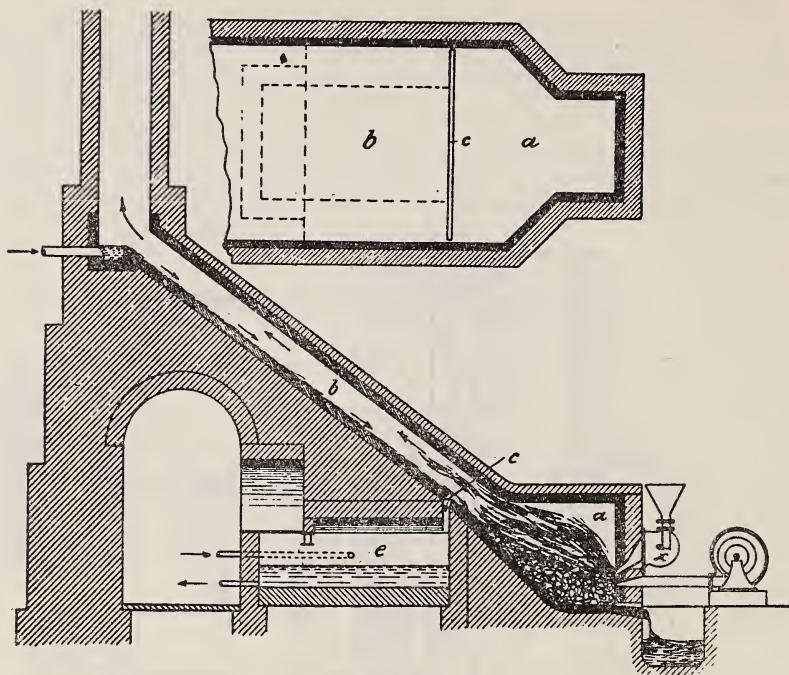


FIG. 21.—Ger. 128903.

Ger. 128903. Schwager, J. Pat. January 25, 1901; Pub. March 11, 1902.

Title: Retort with inclined flue for the evaporation of liquids which develop combustible gases.

The liquid is in direct contact with fuel gas. The gases are lost. The apparatus is more truly an incinerator than an evaporator.

(b) APPARATUS FOR EVAPORATING AND CONCENTRATING WASTE WATER.

Ger. 17869. Ernst, E. Pat. February 8, 1881; Pub. April 17, 1882.

Title: Molasses residues mixed with small coal, peat, shavings, etc.

Complicated retorts or ovens for preparing the mixtures described in the title.

Ger. 20710. Mey, F. H. C. Pat. July 2, 1882; Pub. January 15, 1883.

Title: Apparatus for drying of fruit, sand, brewery waste, and wastes of alcohol plants and sugar factories.

Furnace incorporating the use of an endless belt.

Ger. 24403. Bernard, A. Pat. January 11, 1883; Pub. October 9, 1883.

Title: Apparatus for the evaporation of organic liquids.

An apparatus for the evaporation of organic liquids, especially sugar wastes and elution waters, with the exclusion of air and at the temperature of a hot water bath, consisting of a retort with a stirring and conveying device in combination with a leaching device into which the dried mass arrives out of contact with the air.

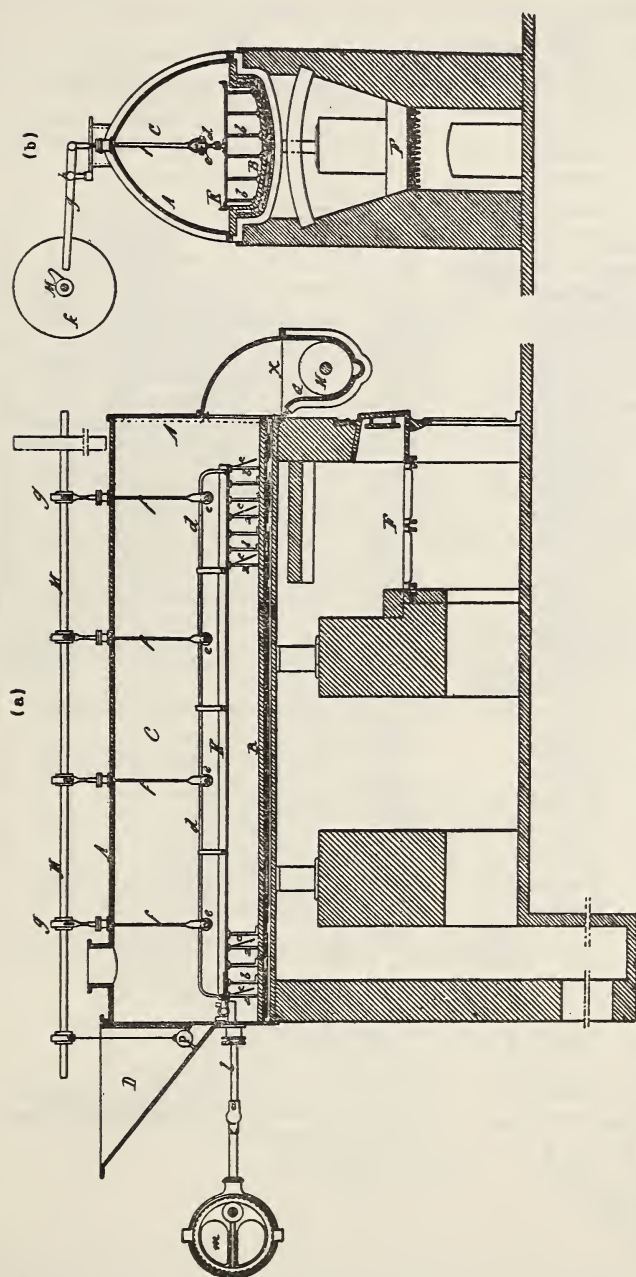


FIG. 22.—Ger. 24403. (See p. 30.)

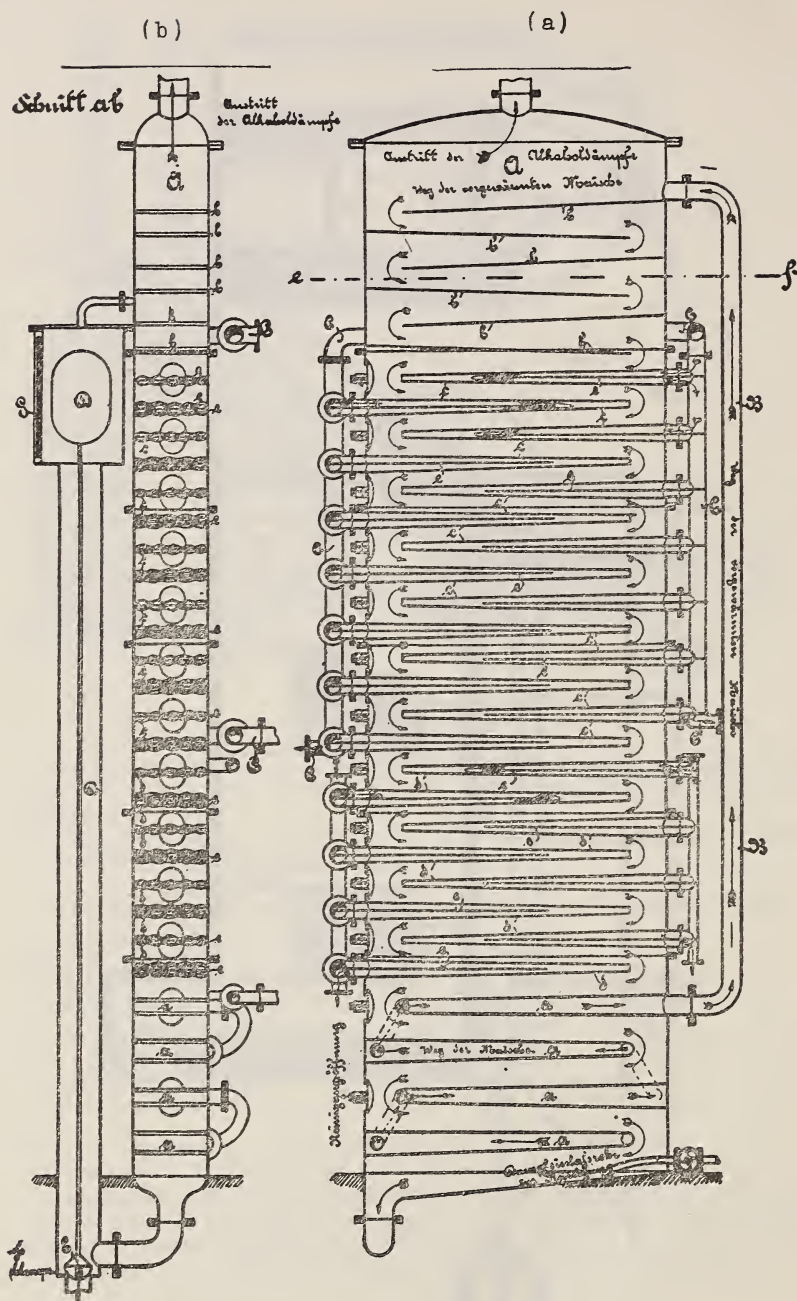


FIG. 23.—Ger. 25945.

Ger. 25945. Salzmann, C. Pat. May 13, 1883; Pub. February 18, 1884.

Title: Mash distilling apparatus for obtaining concentrated waste water.

Ger. 28828 (addition to Ger. 25945). Salzmann, C. Pat. February 22, 1884; Pub. October 1, 1884.

Complicated column still for the recovery of alcohol and concentrated waste water.

Ger. 28249. Plonnis, R. Pat. January 30, 1884; Pub. August 2, 1884.

Title: Apparatus for drying corn, grain, waste water, beet pulp, etc.

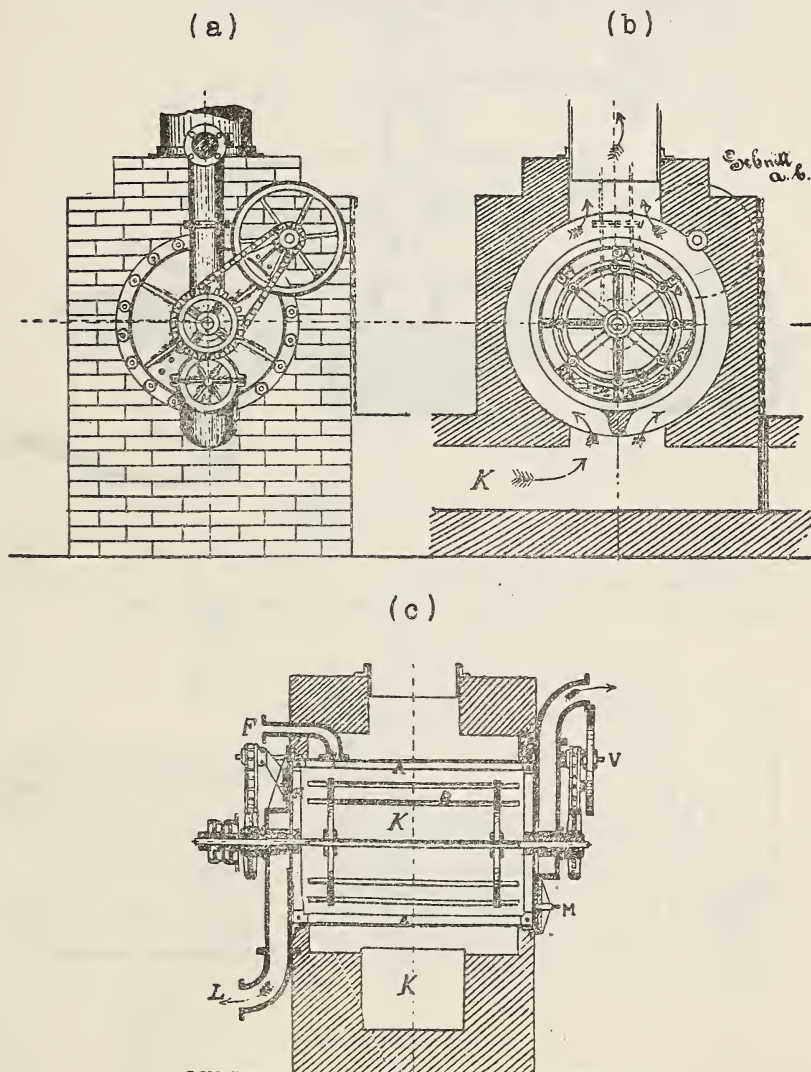


FIG. 24.—Ger. 35853.

Ger. 35853. Klaunig, O. Pat. July 21, 1885; Pub. June 23, 1886.

Title: Vacuum drying apparatus with stirrer for waste water and grain.

Retort with rotating stirrer.

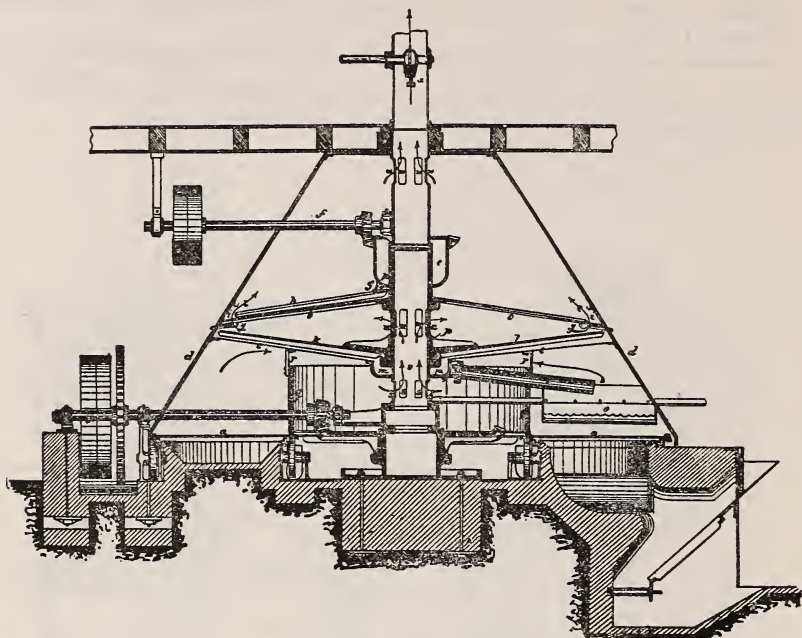


FIG. 25.—Ger. 37641.

Ger. 37641. Wirth & Co. Pat. April 13, 1886; Pub. November 20, 1886.

Title: Drying apparatus for waste water.

Large and complicated circular apparatus rotating about a central shaft.

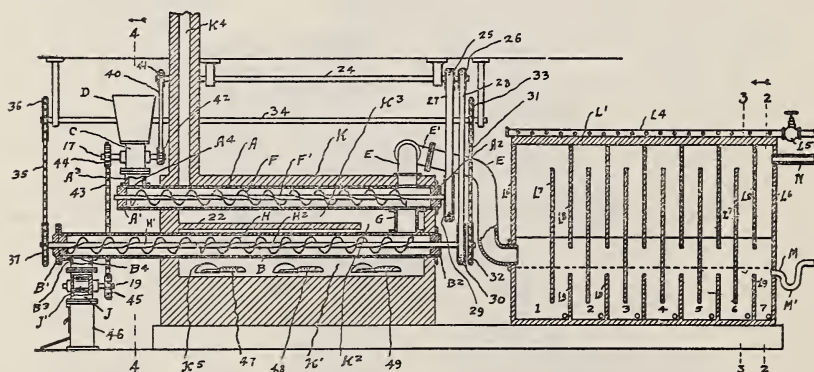
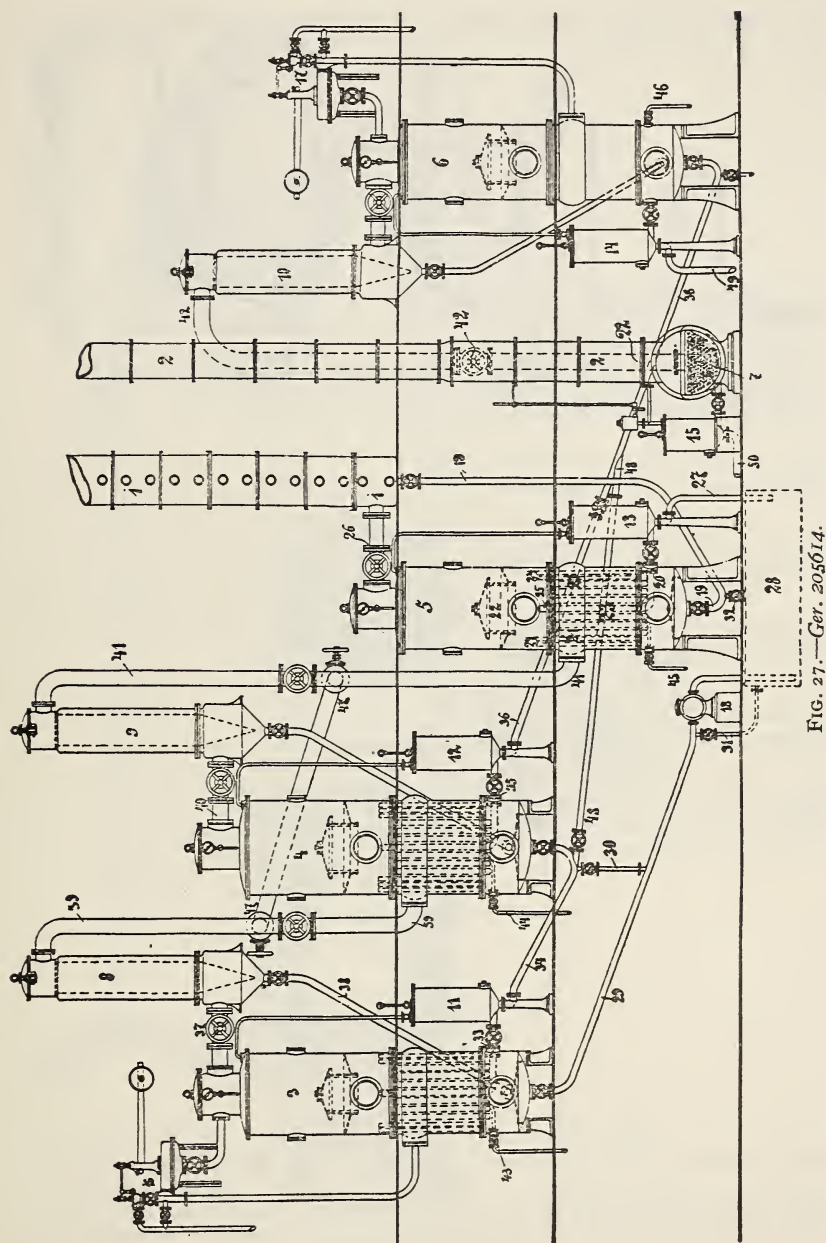


FIG. 26.—U. S. 1379876.

U. S. 1379876. Nottingham and Tuhey, assignors to American Nottingham Process Co. App. May 16, 1919; Pat. May 31, 1921.

Title: Apparatus for distillation.

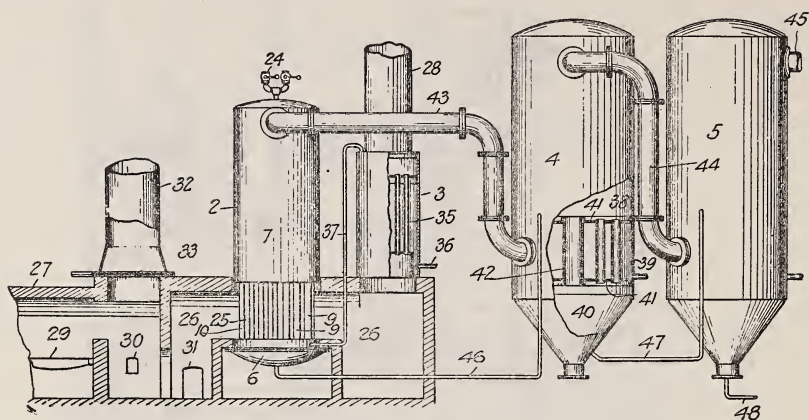
Material, such as corn cobs, is destructively distilled while passing successively through superposed horizontal tubular retorts provided with conveying screws.



Ger. 205614. Pampe, F. Pat. December 2, 1906; Pub. January 15, 1909.

Title: Process for the concentration of waste water.

A diagram gives a complicated plan of three-stage evaporators with various other units for use in the process.



(a)

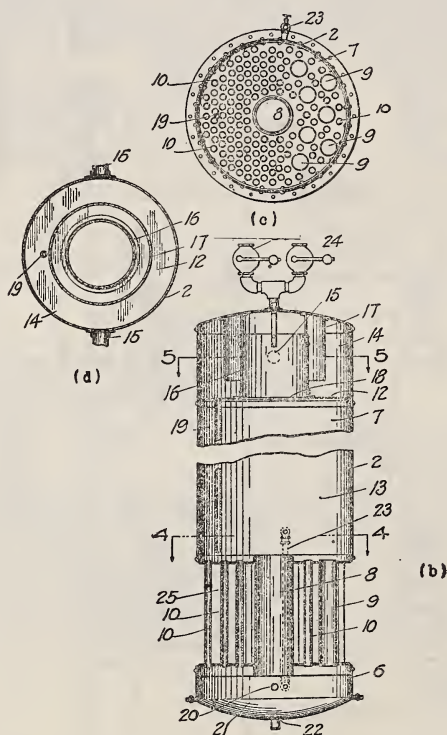


FIG. 28—U. S. 1385499.

U. S. 1385499. Schwarz. App. June 25, 1918; Pat. July 26, 1921.

Title: Evaporator.

For the evaporation of waste water from molasses or similar liquids; a tubular evaporator which may be heated by waste exhaust gases is described.

(c) APPARATUS FOR RECOVERING AMMONIA FROM WASTE WATERS.

(a)

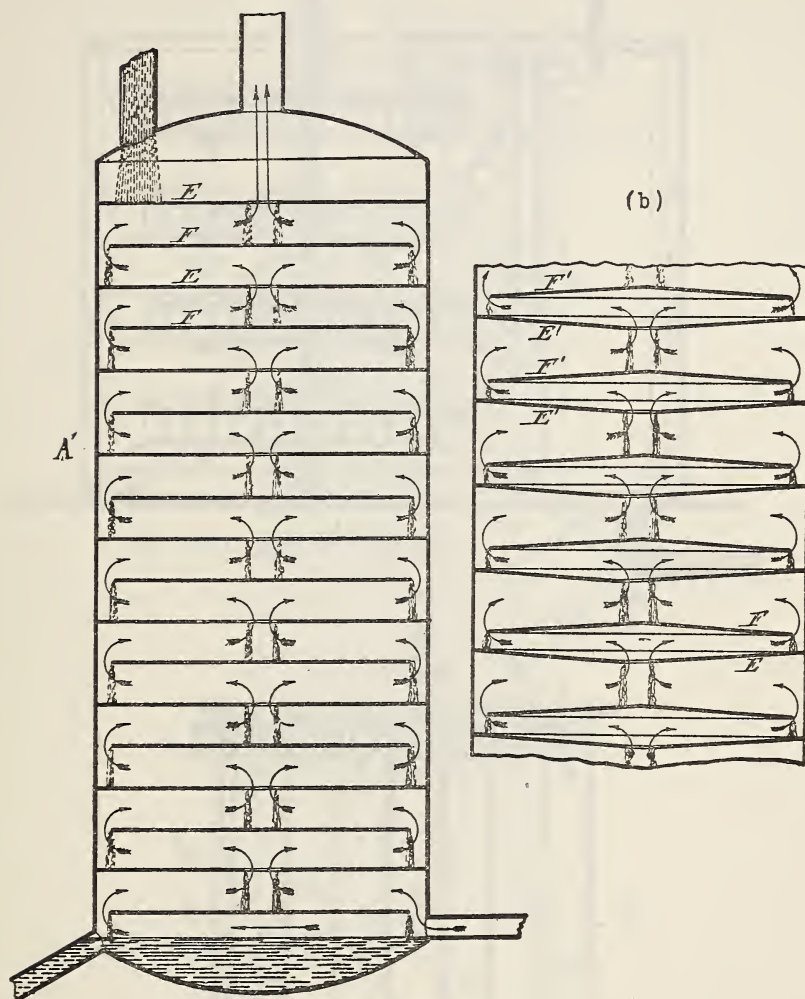


FIG. 29.—Ger. 27034.

Ger. 27034. Young, J. Pat. April 11, 1883; Pub. May 15, 1884.

Title: Apparatus for the recovery of ammonia from water of seal tanks and waste water of sugar factories.

A series of boxes arranged in steps or a tower similar to the modern ammonia still, where ammonia-containing liquids, previously warmed and treated with lime, come in contact with a countercurrent of steam.

Ger. 38596. Schiller, R. Pat. March 12, 1886; Pub. February 18, 1887.

Title: Apparatus for the recovery of ammonia contained in the alcohol or the waste water of the Mannoury molasses desugarizing process.

In the apparatus, ammonium bisulphate is exposed to the gases containing ammonia, changing it to ammonium sulphate. The apparatus is of wood, and composed of plates and arms so arranged to bring the ammonia lyes in contact with the bisulphate solution.

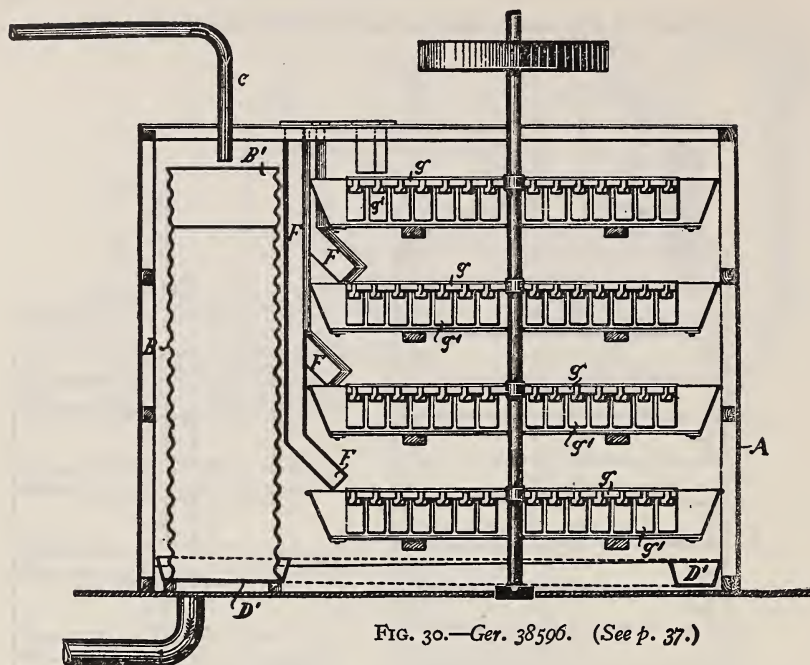


FIG. 30.—Ger. 38596. (See p. 37.)

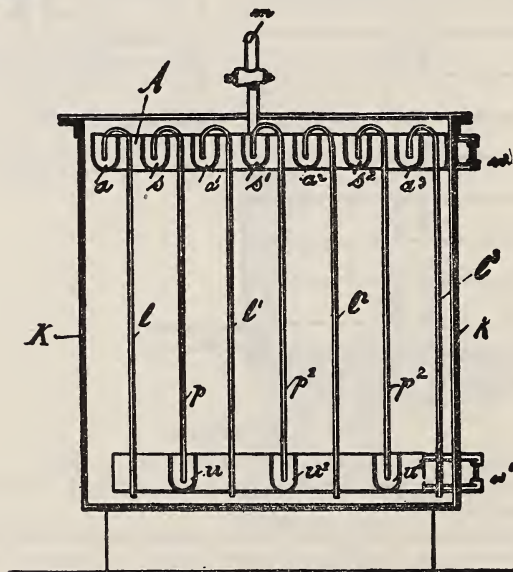


FIG. 31.—Ger. 66465.

Ger. 66465. Mylius, A. Pat. July 19, 1891; Pub. January 2, 1893.

Title: Apparatus for the manufacture of ammonia and other volatile nitrogenous bases from waste waters, etc.

The waste waters and sulphuric acid for absorption of the ammonia are separated by porous walls in a vessel under partial vacuum.

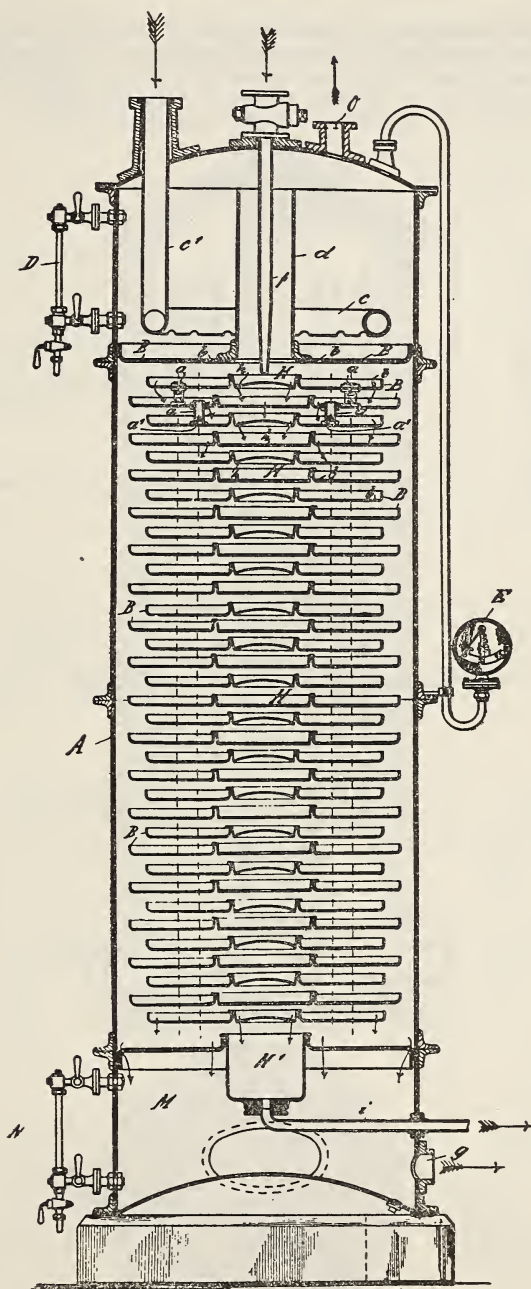
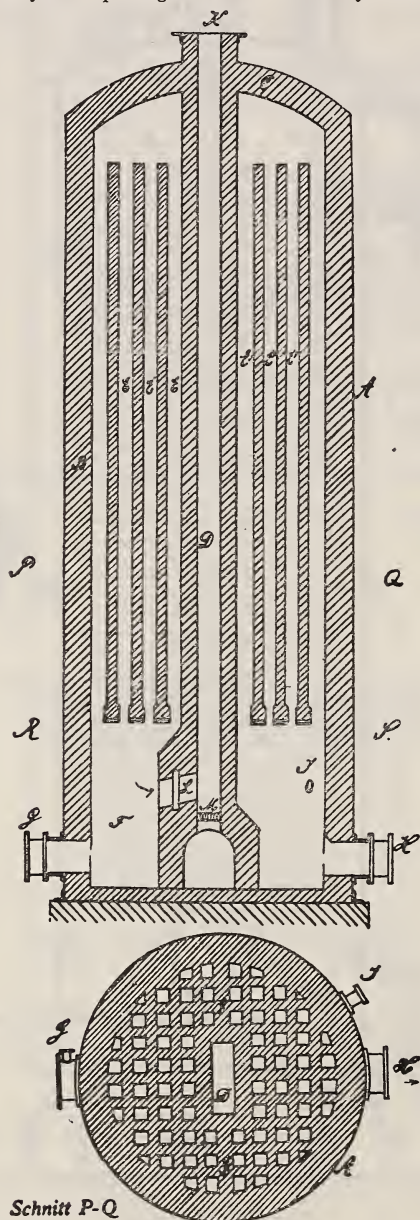


FIG. 32.—Ger. 71414.

Ger. 71414. Seiffert, H. Pat. February 3, 1893; Pub. October 10, 1893.

Title: Apparatus for the recovery of ammonia and other volatile nitrogen bases from waste waters, etc.

The apparatus containing the waste water under vacuum is so constructed that sulphuric acid, when added, falls over a series of plates which have sieve bottoms. The sulphuric acid binds the ammonia under the influence of the vacuum. Dilute sulphuric acid leaves by one opening, the waste water by another.



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FIG. 33.—Ger. 75883.

Ger. 75883. Schulte and Sapp. Pat. August 2, 1893; Pub. July 2, 1894.

Title: Furnace for the manufacture of ammonium cyanide.

Used in the process of manufacturing ammonium cyanide from ammonia and superheated hydrocarbons. A countercurrent system of gases is used. Two ovens make the process continuous.

(d) APPARATUS FOR THE RECOVERY OF POTASH.

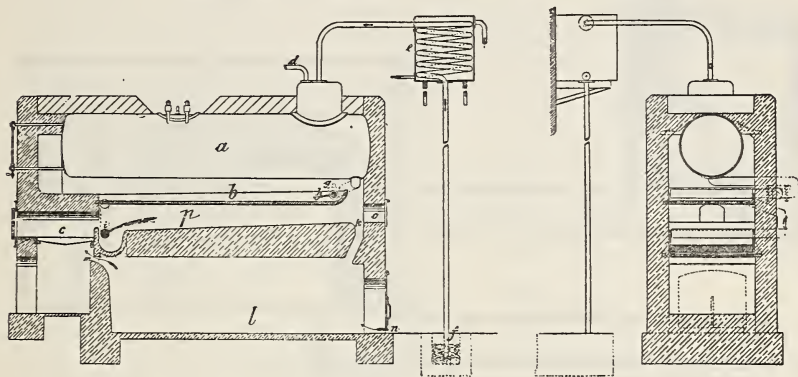


FIG. 34.—Ger. 18493.

Ger. 18493. Roeckner, C. Pat. October 22, 1881; Pub. June 24, 1882.

Ger. 27998. Lutteroth & Co. Pat. November 18, 1883; Pub. July 17, 1884.

Ger. 30724. Lutteroth & Co. Pat. January 24, 1884; Pub. April 13, 1885.

Title: Application of apparatus protected by patent 18493 for the recovery of soda from waste water.

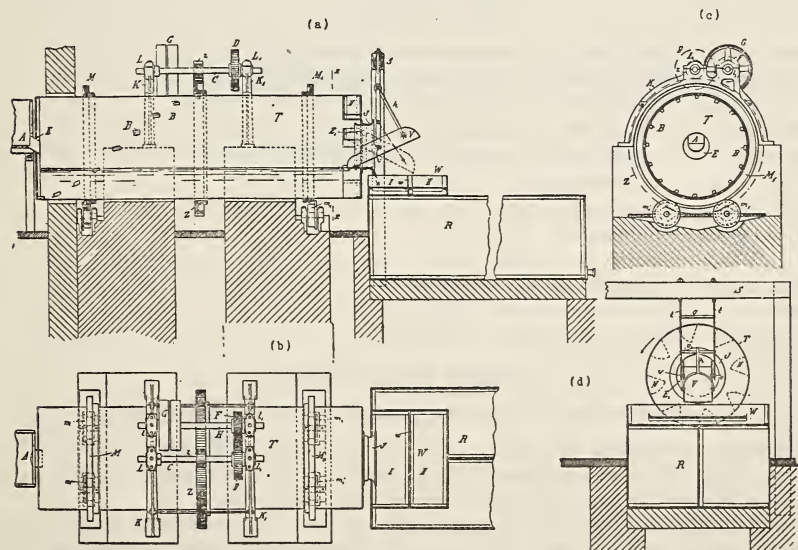


FIG. 35.—Ger. 102900.

Ger. 102900. Recht, H. Pat. June 15, 1898; Pub. May 18, 1899.

Title: Extraction drum for the extraction of waste-water ash.

Similar to rotating drum used in the preparation of milk of lime.

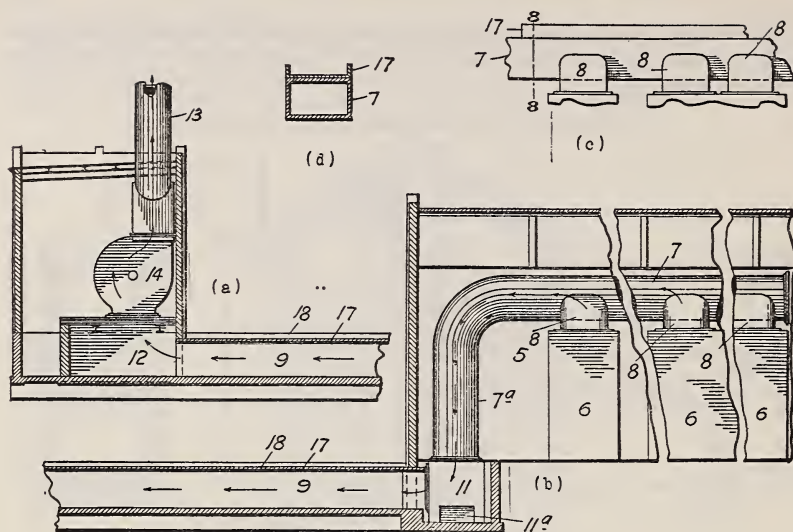


FIG. 36.—U. S. 803058.

U. S. 803058. Henry and Beale, assignors to M. Straus, Denver, Colo. App. Oct. 3, 1904. Pat. Oct. 31, 1905.

Title: Apparatus for extracting crude potash.

Applies to the evaporation of the lyes, which is accomplished by means of troughs built into the flues, thereby utilizing waste heat.

CROSS REFERENCES.

- Ger. 228539. Dieffenbach and Moldenhaver. Group 4 (c).
- Ger. 255073. Dieffenbach and Moldenhaver. Group 4 (c).
- U. S. 247046. Grouven, H. Group 2 (b).
- Fr. 353970. Guerrero, W. de Smirnoff. Group 2 (b).
- Br. 12736-7/11. Guignard and Watrigant. Group 7 (a).
- U. S. 1207416. Kochendoerfer, E. Group 4 (a).
- U. S. 846035. Koppers, H. Group 2 (c).
- U. S. 953960. Koppers, H. Group 2 (c).
- U. S. 488207. Kuntze, P. Group 2 (b).
- U. S. 232991. Lorenzen, H. P. Group 2 (b).
- Ger. 87725. Reichardt and Bueb. Group 4 (a).
- Fr. 331077. Societe F. Bruncke. Group 2 (c).

4. GROUP IV.—CYANIDES.

(a) PRODUCTION OF CYANIDES FROM WASTE WATER DIRECT.

Ger. 86913. Reichardt and Bueb. Pat. October 25, 1894; Pub. May 2, 1896.

Br. 7171/95. Reichardt and Bueb. App. April 8, 1895; Acc. January 4, 1896.

Title: Process for manufacturing cyanides from waste water.

Waste water is distilled out of contact with the air and the gases led through chamotte canals raised to a red or white heat. Cyanides are formed with ammonium carbonate. The gases may be passed through a solution of iron salts to form ferrocyanides. The patentees refer to Ger. 15751 (Group 2(a)), and to the fact that Ortlieb and Mueller (Ger. 9409, Aktien-Gesellschaft Croix, Group 4(b)) were familiar with the production of cyanides from amines.

Ger. 104953. Bueb, J., and Die Dessauer Zuckerraffinerie. Pat. July 27, 1898; Pub. July 27, 1899.

U. S. 642782. Bueb, J., assignor to Roessler & Hasslacher Chemical Co. App. October 1, 1898. Pat. February 6, 1900.

Br. 26259/98. Bueb, J. App. December 12, 1898; Acc. September 30, 1899.

Title: Process of obtaining hydrocyanic acid from gases containing the same.

The steps in the process are as follows: (1) Separating the ammonia contained in the gases, then conducting them through alcohol of high purity and percentage, then subjecting the alcoholic solution of hydrocyanic acid to fractional distillation. (2) Gases from the fractional distillation in (1) pass through a chamber containing alcoholic caustic potash, wherein hydrocyanic acid is separated from the alcohol as an insoluble cyanide. (3) Saturating the mother liquor, which contains hydrocyanic acid, with carbon dioxide (contained in the gas mixture), whereby the alkali will be precipitated as a carbonate, while hydrocyanic acid becomes dissolved in the alcohol and may be separated therefrom.

Fr. 296793. Bueb, J. Pat. February 2, 1900.

Title: Process of cyanizing gases from vinasses. (Unpublished.)

Ger. 113530. Bueb, J. Pat. August 20, 1899; Pub. August 13, 1900.

Title: Process of manufacturing cyanogen compounds from waste water and similar gases according to patent 86913.

In the older process, the retorts became congested with carbon after a period of heating. This patent provides for alternating the ovens while this carbon deposit is burned out.

Ger. 120264. Besemfelder, E. Pat. May 19, 1900; Pub. April 30, 1901.

Br. 4323/01. Besemfelder, E. App. February 28, 1901; Acc. October 19, 1901.

Title: Manufacture of cyanogen compounds from gas mixtures containing ammonia.

The ammoniacal gases produced by the destructive distillation of organic nitrogenous matter are passed through a rope or chain tower fed with cold, strongly alkaline liquid, whereby ammonium salts are decomposed with the formation of ammonia. The gases are then dried by passing through scrubbers containing caustic lime before being led to the cyanizing generators. These contain incandescent coke, assumed to have been in previous use. These generators are blown hot and the gas passed through. They are arranged in series and are equipped with carbon feed and ash pits.

Ger. 181508. Chemische Fabrik Schlempe. Pat. January 17, 1906; Pub. February 16, 1907.

Fr. 367502. Chemische Fabrik Schlempe.

Title: Process for the manufacture of cyanogen compounds from molasses waste water.

Tar produced in the destructive distillation of waste water containing nitrogen is partially burned with access of air and passed through highly heated tubes to produce cyanogen. Also, the tar may be distilled without access to the air, mixing the resulting gases with water gas and passing through the hot tubes. Refers in the patent to Ger. 86913 and 113530.

Ger. 232615. Chemische Fabrik Schlempe. Pat. February 16, 1910; Pub. March 16, 1911.

U. S. 1032988. Bueb, J. App. October 26, 1910; Pat. July 16, 1912.

Title: Process for obtaining cyanogen and its compounds.

The cyanizable and noncyanizable gases from waste water distillation are separated before cyanizing; for example, by condensation, between the retort and superheater, or by counter-current scrubbing of the gases with condensate to remove all cyanizable substances. The scrubbing solution is then distilled and the gases passed into the superheater.

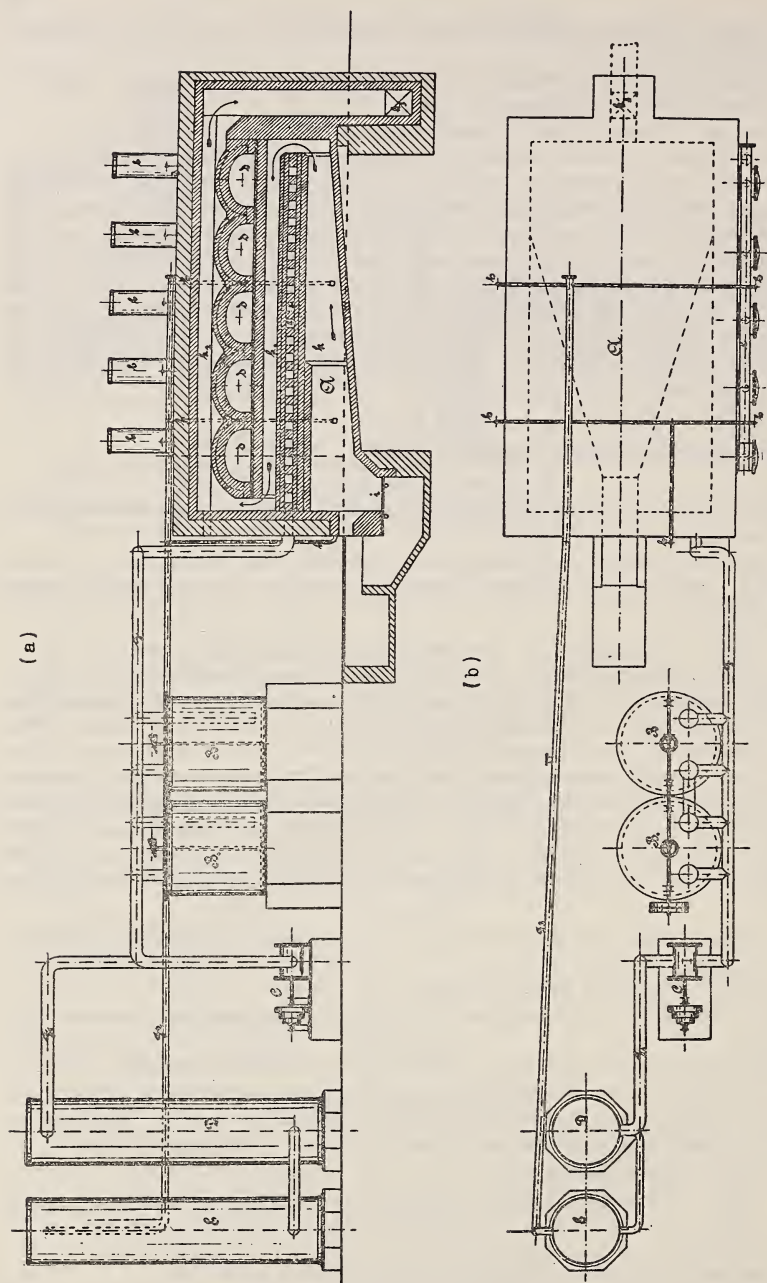


FIG. 37A.—Ger. 87725.

Ger. 87725 (addition to Ger. 86913). Reichardt and Bueb. Pat. September 29, 1895; Pub. July 18, 1896.

Title: Furnace for the manufacture of cyanogen compounds from waste water.

Description of the furnace used for the process described in 86913. (See p. 42.)

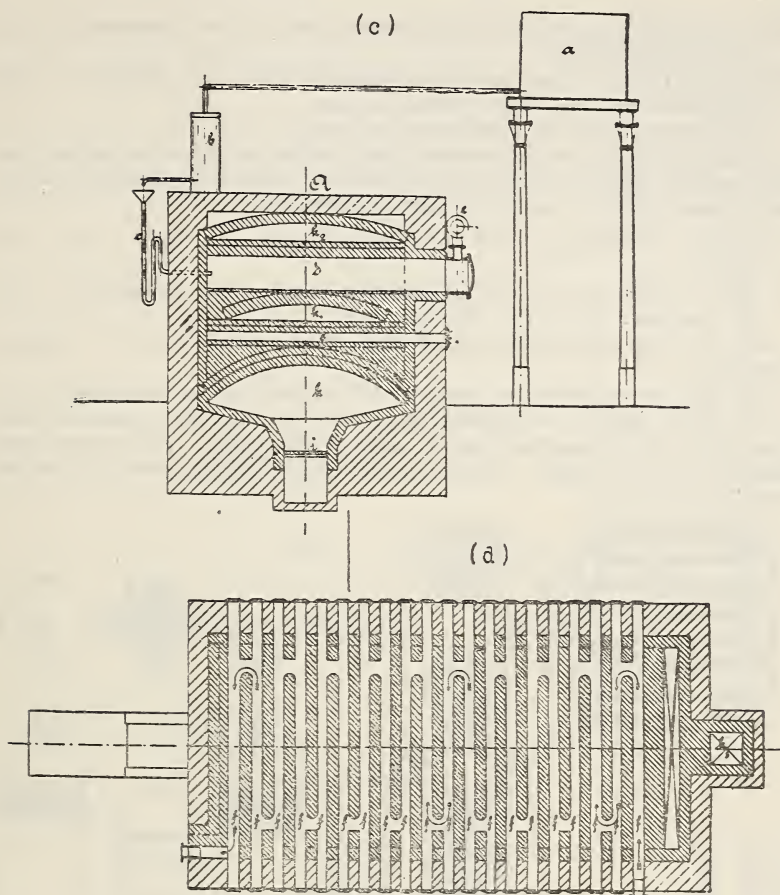


FIG. 37B.—Ger. 87725.

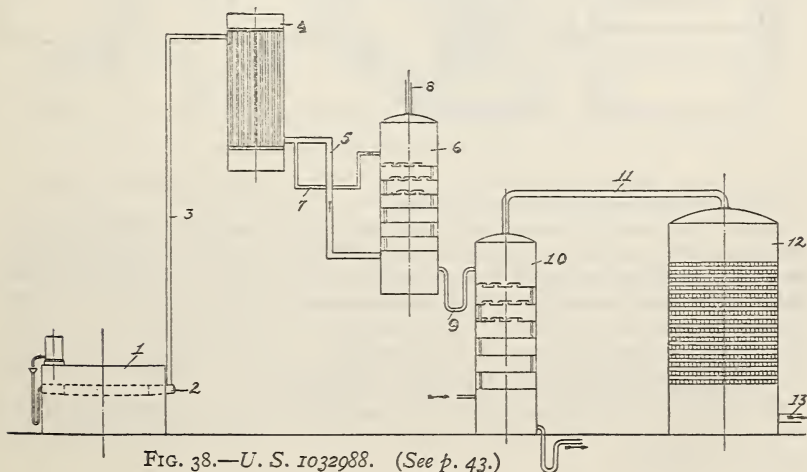


FIG. 38.—U. S. 1032988. (See p. 43.)

Ger. 255440. Deutsche Gold und Silberscheide-Anstalt. Pat. November 9, 1911; Pub. January 3, 1913.

Fr. 447725. Deutsche Gold und Silberscheide-Anstalt. App. August 29, 1912; Pat. November 5, 1912; Pub. January 13, 1913.

Title: Process of cyanizing gases from waste water.

The gases are superheated in chambers, free of baffling substances, by radiant heat. They are then passed through hot tubes which are smooth, thick and nonporous, such as fused quartz or a mixture of quartz and zirconia.

Ger. 259501. Deutsche Gold und Silberscheide-Anstalt. Pat. April 7, 1912; Pub. April 25, 1913.

Fr. 448722. Deutsche Gold und Silberscheide-Anstalt. App. September 5, 1912; Pat. December 4, 1912; Pub. February 8, 1913.

Title: Process of obtaining cyanogen from the gases from waste water or analogous substances.

Gases from the calcination of waste water are superheated in refractories of acid nature without regard to the physical properties of porosity, etc., choosing a speed of flow which will suppress the catalytic action of the surfaces. This process avoids separation of the tar formed. The gases are led directly from the retort to the super-heater.

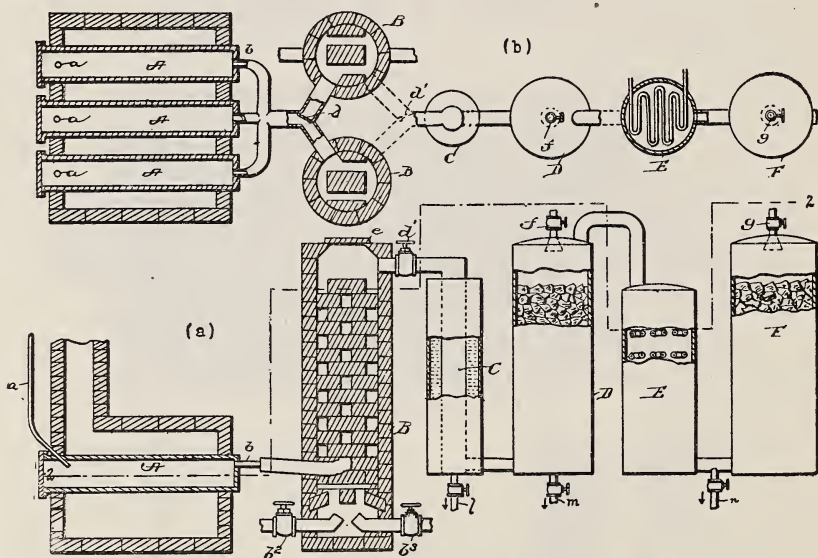


FIG. 39.—U. S. 1145484.

U. S. 1145484. Huber and Poindexter. App. July 21, 1914; Pat. July 6, 1915.

Title: Utilizing beet molasses waste water.

Steffen waste water is concentrated and destructively distilled in the absence of air, the gases are superheated, changing some to hydrocyanic acid. The ammonia is absorbed in sulphuric acid and the hydrocyanic acid in water. Gives the following yields: 55 per cent of nitrogen obtained as hydrocyanic acid; 26 per cent of nitrogen obtained as ammonia; and 90 per cent of potassium obtained as salts.

U. S. 1165358. Kochendoerfer, E. App. October 31, 1912; Pat. December 21, 1915.

Title: Process for manufacturing nitrogen compounds.

For producing cyanides from waste water superheaters are used, composed of silica, zirconia, etc., with smooth unimpeded walls and free passages. Heating is accomplished by radiation.

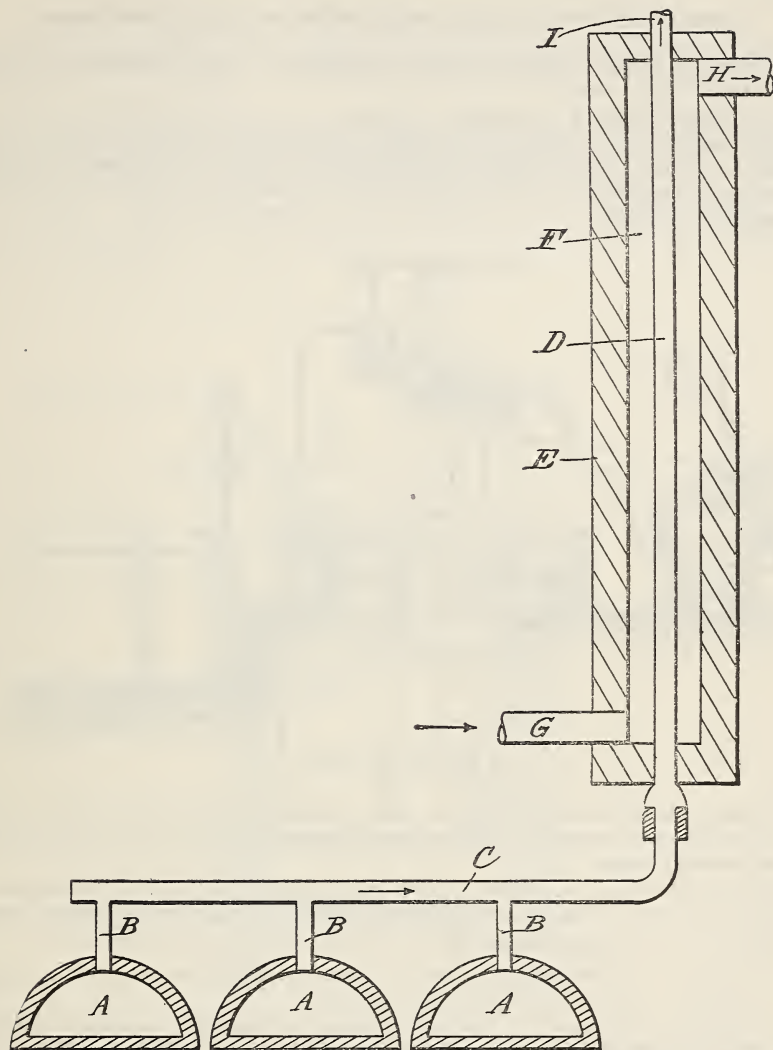


FIG. 40.—U. S. 1165358. (See p. 46.)

U. S. 1207416. Kochendoerfer, E. App. March 26, 1913; Pat. December 5, 1916.

Title: Process of manufacturing nitrogen compounds.

The patent claims that for producing cyanides from waste water, rough or porous surfaces are deleterious; claims use of high speed of gases in superheating; for example, 7 m. per second for pyridines; 10 m. per second for amino compounds. It is necessary to use material of an acid nature for the superheaters, for instance, Dinas stone (chiefly quartz), but basic materials may also be used if the speed is high enough. The distillation gases from waste water incineration are used directly without separation of the inactive constituents.

(b) PRODUCTION OF CYANIDES FROM AMINES BY SPECIAL PROCESSES.

Br. 3844/79. Societe Anonyme de Croix and Imbray, J. App. September 24, 1879; Acc. March 5, 1880.

Title: Manufacture of hydrocyanic acid, etc.

The methylamine of commerce is vaporized and the vapors passed through a retort heated to redness, the products being absorbed in sodium or potassium hydroxide and the ammonia in acid. The methylamine vapors may also be passed into molten potassium hydroxide.

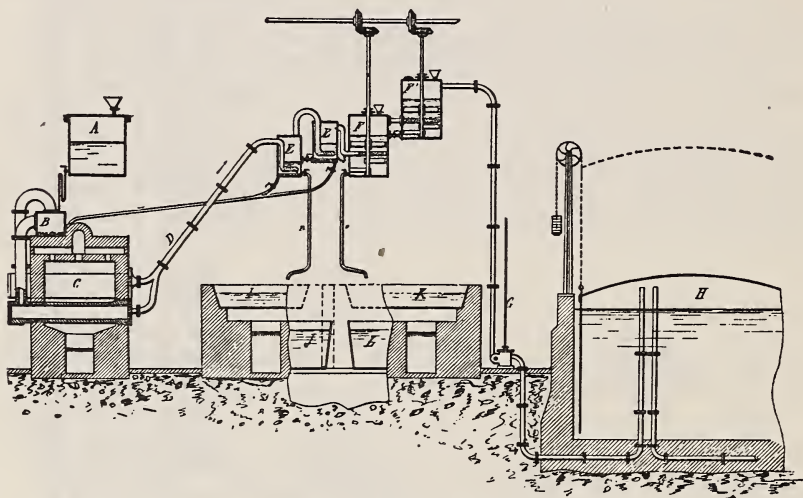


FIG. 41.—Ger. 9409.

Ger. 9409. Aktien-Gesellschaft Croix. Pat. September 27, 1879; Pub. April 5, 1880.

Title: Improved process of manufacturing hydrocyanic acid and its derivatives from trimethylamine.

This patent covers the original Ortlieb and Mueller process for the production of hydrocyanic acid. If gaseous trimethylamine is led through glowing vessels, great quantities of hydrocyanic acid are formed, as well as ammonia and fuel gas.

U. S. 967943. Liebknecht, O., assignor to Roessler & Hasslacher Chemical Co. App. April 16, 1910; Pat., August 23, 1910.

U. S. 969885. Liebknecht, O., assignor to Roessler & Hasslacher Chemical Co. App. February 28, 1910; Pat. September 13, 1910.

Br. 9375/10. Deutsche Gold und Silberscheide-Anstalt. App. April 18, 1910; Acc. October 27, 1910.

Title: Improvements in the manufacture of sodium cyanide.

Trimethylamine may be directly decomposed with the formation of sodium cyanide by passing the dry gas through molten metallic sodium. A hydrocarbon, carbon and hydrogen being formed at the same time. The sodium cyanide may be separated from the carbon by filtration, or the formation of carbon may be avoided by the addition of disodium cyanamide, or by allowing ammonia to pass into the fused metallic sodium along with the trimethylamine. The temperature of the molten sodium should be about 700° C.

(c) PRODUCTION OF CYANIDES FROM VARIOUS GAS MIXTURES.

Br. 3735/93. de Lambilly, P. R. App. February 20, 1893; Acc. November 11, 1893.

Title: An improved method of producing ammonium formate from ammonia and from this, hydrocyanic acid.

A mixture of carbon monoxide and ammonia passed over a porous substance at $100-130^{\circ}\text{C}$. produces ammonium formate, which, when passed through a set of tubes at 210°C . in which is more porous substance forms hydrocyanic acid and water.

Br. 26236/97. Lance and Bourgade. App. November 11, 1897; Acc. July 2, 1898.

Title: A Process for the economic production of cyanides, their derivatives and other nitrogen compounds.

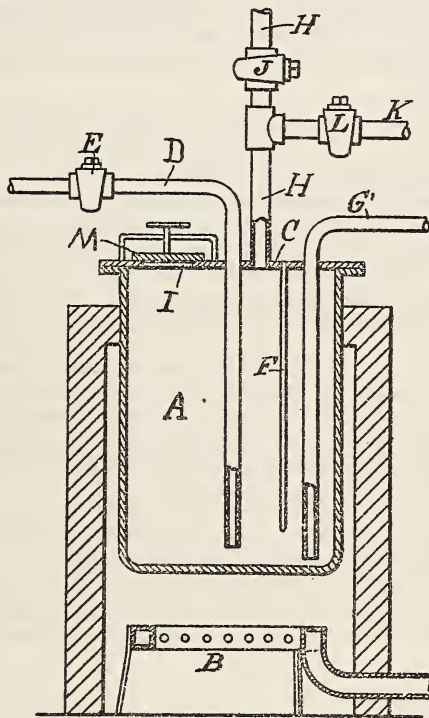


FIG. 42.—U. S. 969885. (See p. 48.)

On passing a mixture of nitrogen, hydrogen, and ammonia over intensely heated carbon, ammonium cyanide is formed and volatilized. To obtain the maximum yield, an excess of hydrogen must be used. Instead of pure hydrogen, hydrocarbons may be used which are obtained from the distillation of organic matter, and nitrogen as in the air. A typical mixture is 2,000 l. of hydrocarbon, 200 l. of nitrogen, and 80 l. of ammonia passed through carbon heated to $1,100^{\circ}\text{C}$. The ammonium cyanide is absorbed by an alcoholic washer. Sodium or potassium cyanide may be obtained by treating with an alcoholic solution of soda or potash.

Br. 19804/02. Woltreck, H. C. App. September 10, 1902; Acc. September 10, 1903.

Title: Process for producing hydrocyanic acid.

Perfectly dry ammonia and a volatilized or gaseous carbon compound, also perfectly dry, are passed together with hydrogen in equal volumes, over a strongly heated catalytic agent, such as platinized pumice. One volume of ammonia and two volumes of water gas make a convenient mixture. The hydrocyanic acid is absorbed, preferably in an alkaline solution.

Ger. 229057. Dieffenbach and Moldenhaver. Pat. June 12, 1908; Pub. November 28, 1910.

U. S. 954080. Dieffenbach and Moldenhaver. App. April 16, 1908; Pat. April 5, 1910.
Title: Process for producing hydrocyanic acid.

Hydrocyanic acid is produced by passing a mixture of hydrogen, nitrogen, and not over 40 per cent carbon monoxide or dioxide over highly heated coal.

Ger. 228539. Dieffenbach and Moldenhaver. Pat. February 27, 1908; Pub. November 10, 1910.

Ger. 255073 (addition to Ger. 228539). Dieffenbach and Moldenhaver. Pat. July 4, 1911; Pub. December 27, 1912.

Title: Hydrocyanic acid from nitrogen and hydrocarbons in an electric arc.

Gases are passed through a resistance furnace, composed of a coke floor with adjustable electrodes. Provision is made for the introduction of fresh coke, thereby making the process continuous.

Br. 18945/09. Beindl, C. App. August 17, 1909; Acc. August 17, 1910.

U. S. 1144457. Beindl, C. App. September 1, 1910; Pat. June 29, 1915.

Title: Method of producing cyanogen compounds.

Cyanides are obtained from ammonia and volatile or gaseous carbide by passing the mixture over a catalyser in the form of wire net, heated to the appropriate temperature, of one of the following substances: Gold, silver, iridium, palladium, rhodium, copper, manganese, cobalt, nickel, iron, chromium, and their nitrides; also such alloys as copper-zinc. The gases used must be pure and dry. For example, ammonia and acetylene passed over copper forms hydrocyanic acid at about 480° C.

Br. 7260/15. Beindl, C. App. May 14, 1915; Acc. August 16, 1917.

Br. 10321/15. Beindl, C. App. July 15, 1915; Acc. July 11, 1921.

Title: An improved process for the production of hydrocyanic acid.

Specifies, in addition to the catalysts given in Br. 18945/09, etc., the following: Nitrides of the ores of molybdenum, uranium, tungsten, osmium, titanium, and possibly the oxides, produced at a temperature of 700° C.

Ger. 232878. Hauff. Pat. August 3, 1910; Pub. March 25, 1911.

Title: Process for changing hydrocyanic acid to ammonia.

Gases containing hydrocyanic acid are mixed with steam as they come from the electric furnace (at 1,000–1,300° C.), and the current of gas is passed through a bundle of tubes of fire-resisting porcelain or the like which is maintained at the proper temperature.

U. S. 1387170. Poindexter, R. W. App. July 6, 1920; Pat. August 9, 1921.

Title: Process of producing hydrocyanic acid.

The process consists of heating a hydrocarbon and injecting ammonia into it, further heating to cause reaction, above 500° C., and again heating to 1,000° C. The upper limit is specified as 1,450° C.

(d) RECOVERY OF HYDROCYANIC ACID FROM GAS MIXTURES.

Br. 1608/08. Percy and Byrom. App. January 23, 1908; Acc. December 17, 1908.

Title: Improvements in obtaining or extracting hydrocyanic acid from waste gases.

Hydrocyanic acid is recovered by a counter-current washing in a tower with a trickle of water so as not to absorb sulphur. The sulphur may be precipitated with lead, and the hydrocyanic acid recovered as usual by distillation.

Ger. 243469. Guignard and Watrigant. Pat. January 21, 1911; Pub. February 13, 1912.

Fr. 436185. Guignard and Watrigant. App. January 16, 1911; Pat. January 17, 1912; Pub. March 20, 1912.

Title: Process of recovery of hydrocyanic acid contained in a gas.

Hydrocyanic acid may be absorbed in a solution of an alkali dissolved in alcohol, and destroying, eliminating, or absorbing the water formed in the reaction. This may be done by adding from time to time metallic sodium.

Fr. Addition No. 15080. Guignard and Watrigant. Pat. February 10, 1911.

The alcohol in the above patent specification may be replaced with glycerine.

In addition to the patents listed above, the reader is also referred to the many patents wherein the methods of recovery of the hydrocyanic acid are incorporated with the other details of the process, as in the preceding subgroups.

(e) PREPARATION OF CYANIDES IN MARKETABLE FORM.

U. S. 716350. Roessler, F., assignor to Roessler & Hasslacher Chemical Co. App. February 4, 1899; Pat. December 16, 1902.

Title: Method of making sodium cyanide.

The absorbing caustic solution is maintained at a temperature above 33° C., for instance, at 50° C., so that anhydrous sodium cyanide is precipitated when the concentration of sodium cyanide becomes greater than 517 g. per liter.

Ger. 192884. Chemische Fabrik Schlempe. Pat. August 28, 1906; Pub. December 14, 1907.

U. S. 859482. Bueb, J., assignor to Roessler & Hasslacher Chemical Co. App. March 19, 1907; Pat. July 9, 1907.

Title: Cyanide briquet and process for making same.

The cyanide is melted and free caustic hydroxide is added. Both crystallize together and are dried and briquetted.

Ger. 194446. Chemische Fabrik Schlempe. Pat. April 27, 1907; Pub. January 20, 1908.

U. S. 912538. Bueb, J., assignor to Roessler & Hasslacher Chemical Co. App. April 15, 1908; Pat. February 16, 1909.

Title: Process of making cyanide briquets.

After briquetting alkali cyanides they are allowed to stand for several hours in a closed room under a high vacuum at a temperature of 70° C., thereby driving off the moisture without decomposing the cyanide.

U. S. 1232471. Abegg, F., assignor to Roessler & Hasslacher Chemical Co. App. June 23, 1914; Pat. July 10, 1917.

Title: Cyanide in the form of granules.

Molten cyanide is projected in a thin jet against a plate so as to form hollow droplets (most of which are open at one end) when solidified, and glazed both inside and outside.

U. S. 1262057. Kaufmann, A., assignor to Air Reduction Co. App. November 7, 1916; Pat. April 9, 1918.

Title: Sodium cyanide briquet.

Granular sodium cyanide or the like is compressed into a briquet with two oppositely placed indentures to facilitate breaking and with two supporting ribs to provide free space about the briquet. The briquet thus formed is passed through a suitable furnace to fuse the outer surface, thus providing a protective coating. The patent claims easy solubility by reason of the inner granular condition, and high resistance to absorption of water and carbon dioxide by reason of the protective coating.

CROSS REFERENCES.

Fr. 59268. Evrard. Group 7 (d).

Ger. 339302. Muhlert. Group 2 (a).

5. GROUP V.—NITROGENOUS NONSUGARS.

(a) BETAINE AND GLUTAMINIC ACID FROM WASTE WATER AND MOLASSES.

Fr. 344954. Stiepel, C. App. July 19, 1904; Pat. September 23, 1904; Pub. November 18, 1904.

Ger. 157173. Stiepel, C. Pat. March 4, 1904; Pub. December 9, 1904.

Br. 15934/04. Stiepel, C. App. July 18, 1904; Acc. May 18, 1905.

Title: Process for extracting betaine or its salts from molasses.

Concentrated molasses is mixed with twice its volume of 95 per cent alcohol. The mixture is allowed to settle, the alcoholic layer decanted off, filtered, and decolorized with animal charcoal. It is concentrated to a syrup and treated with strong hydrochloric acid and cooled. Crystals of betaine hydrochloride which separate out are purified by recrystallization from alcohol.

Ger. 243332. Stolzenberg, H. Pat. May 17, 1911; Pub. February 13, 1912.

Fr. 443909. Stolzenberg, H. App. May 14, 1912; Pat. July 25, 1912; Pub. October 5, 1912.

Title: Process of manufacture of betaine hydrochloride from molasses, waste water and other residues of beet-sugar manufacture.

In the production of betaine hydrochloride from molasses and other waste from beet-sugar manufacture, the initial material is saturated with gaseous hydrochloric acid, the humus substances resulting from the decomposition of the sugar are filtered off from the hydrochloric-acid solution, which is allowed to stand in the cold. The precipitated alkali chlorides and the glutaminic acid hydrochloride are separated from the betaine hydrochloride remaining in the solution, the solution is evaporated in vacuo to a sirupy consistency and alcohol is added.

Ger. 280824. Melasseschlempe Gesellschaft. Pat. December 18, 1912; Pub. November 28, 1914.

Title: Manufacture of glutaminic acid and alkali chlorides from molasses waste water, molasses, or other residuals of beet-sugar manufacture.

After the recovery of betaine, according to Ger. 243332, the glutaminic acid is recovered in the residue. The mixture of chlorides is treated with alcohol, then gaseous hydrochloric acid. Glutaminic acid hydrochloride forms, which is very soluble. The alkali chlorides are insoluble in alcohol and are filtered off and the residual liquor evaporated and saponified by boiling with water. Further evaporation gives the glutaminic acid, which may be further purified.

Ger. 264390. Hofmann, LaRoche & Co. Pat. February 22, 1913; Pub. September 13, 1913.

Title: Process for the manufacture of an iron salt of glutaminic acid.

When aqueous solutions of glutaminic acid are heated with metallic iron, air being excluded, ferrous salts of glutaminic acid are obtained. Aqueous glutaminic acid solutions have no action on ferric oxide or sulphide.

Ger. 276489. Aktien Gesellschaft fur Anilin Fabrikation. Pat. May 8, 1913; Pub. July 10, 1914.

Br. 2813/13. Aktien Gesellschaft fur Anilin Fabrikation. App. May 7, 1913; Acc. April 23, 1914.

Br. 2923/13. Aktien Gesellschaft fur Anilin Fabrikation. App. October 16, 1913; Acc. June 4, 1914.

Fr. 468366. Aktien Gesellschaft fur Anilin Fabrikation. App. February 12, 1914; Pat. April 22, 1914; Pub. July 4, 1914.

U. S. 1219226. Bergami and Saemann, assignors to Aktien Gesellschaft fur Anilin Fabrikation. App. March 4, 1914; Pat. March 13, 1917.

Title: Process for the manufacture of betaine from vinasses.

Crude betaine hydrochloride is obtained by treating a vinasse, nearly dehydrated by evaporation in a vacuum, with sufficient hydrochloric acid to produce a strongly acid reaction to Congo indicator, keeping the temperature below 60° C., cooling, filtering, and washing the crystals obtained with a little alcohol, and drying. On recrystallizing from methyl alcohol, a product of 95 per cent purity may be obtained, which may be converted into white betaine hydrochloride by crystallizing from water in the presence of animal charcoal. By using hydrobromic or hydriodic acids instead of hydrochloric, the corresponding bromide or iodide may be obtained.

Fr. 483313. Corti, A. App. October 31, 1916; Pat. March 26, 1917; Pub. June 26, 1917.

Title: Process for separating glutaminic acid from other amino acids.

Excess of free hydrochloric acid prevents glutaminic acid from precipitation. The acid solution is cooled and exactly neutralized. Glutaminic acid is 1 per cent soluble. It crystallizes in fine grain, and is then recrystallized. An example is given of its preparation from wheat gluten. Other amino acids are left in the mother liquors.

(b) BETAINE PREPARED FROM OTHER SOURCES.

Ger. 269701. Aktien Gesellschaft fur Anilin Fabrikation. Pat. October 25, 1912; Pub. January 28, 1914.

Br. 15162/13. Aktien Gesellschaft fur Anilin Fabrikation. App. July 1, 1913; Acc. September 11, 1913.

Title: Process for manufacturing betaine and its salts.

A quantitative yield of betaine is said to be obtained by the action of trimethylamine on an alkali salt of monochloroacetic acid, preferably in aqueous solution and under pressure. The product may be evaporated to dryness and the betaine extracted from the residue by means of absolute alcohol. To prepare a salt of betaine, the necessary acid is added to the liquid before or during evaporation.

Ger. 269751. Aktien Gesellschaft fur Anilin Fabrikation. Pat. October 17, 1912; Pub. January 30, 1914.

Br. 15161/13. Aktien Gesellschaft fur Anilin Fabrikation. App. July 1, 1913; Acc. August 28, 1913.

Fr. 459561. Aktien Gesellschaft fur Anilin Fabrikation. App. July 21, 1913; Pat. September 10, 1913; Pub. November 8, 1913.

Br. 16710/13. Aktien Gesellschaft fur Anilin Fabrikation. App. July 21, 1913; Acc. September 4, 1913.

Title: Process for making betaine hydrochloride.

The hydrochloride of the methyl ester of aminoacetic acid is converted into betaine hydrochloride by heating with methyl alcohol. Other derivatives of aminoacetic acid may be used, followed by saponification.

6. GROUP VI.—POTASH AND ALKALI SALTS.

Fr. 167119. Lesage-Montagne. Pat. February 21, 1885.

Title: Leaching and exhausting salts from beets and potash from wool fat. (Unpublished.)

Ger. 77182. Societe Wache Locoge et Cie. Pat. September 1, 1893; Pub. October 10, 1894.

Title: Process of refining beet potash.

Process adapted for French potash lyes. The raw potash is leached with a concentrated solution of potassium sulphate or chloride, depending upon which salt predominates.

Ger. 88003. Graff, W. Pat. February 23, 1896; Pub. July 22, 1896.

Title: Separation of sodium hydroxide from solutions of potassium and sodium hydroxides as potassium sodium carbonate.

Sufficient concentrated solution of potassium carbonate is added to a solution containing potassium and sodium hydroxides to form a double salt, which separates in crystals upon evaporation. A solution of waste-water ash is made caustic with lime, evaporated to 50° B. to separate potassium sulphate and potassium chloride, and then freed in the above manner from sodium hydroxide.

Ger. 91730. Graff, W. Pat. October 31, 1896; Pub. April 3, 1897.

Title: Separation of sodium hydroxide from solutions of potassium and sodium hydroxides as potassium sodium carbonate.

The solution from the main patent, No. 88003, already contains potassium carbonate and sodium hydroxide. This is evaporated almost to dryness to separate potassium sodium carbonate from lime. The residue is extracted with alcohol which dissolves potassium hydroxide, and the potassium sodium carbonate remains behind.

Fr. 312562. Savary. Pat. July 6, 1901.

Title: New process for the treatment of molasses vinasses for the extraction of potassium salts and glycerine, and the utilization of nitrogen. (Unpublished.)

Ger. 191105. Bauer, E. Pat. December 4, 1906; Pub. November 9, 1907.

Fr. 372415. Bauer, E. App. December 11, 1906; Pat. February 18, 1907; Pub. April 6, 1907.

Title: Process of manufacture of a high-percentage technically pure potash salt from beet-sugar waste salts.

Potash of high percentage is obtained by purifying the liquors, concentrating to 58–62° B., raising the temperature above 90° C., when potassium sodium carbonate, which contains nearly all the soda, separates. This precipitate is separated. The remaining liquor is heated to 90° C. and cooled to about 50° C. when a new precipitate is formed, which is separated, and the sodium chloride, containing potassium chloride, is diluted to 52° B. and cooled to precipitate potassium chloride.

Ger. 210273. Grun, E. Pat. November 10, 1906; Pub. May 25, 1909.

Title: Process of manufacture of high-percentage potash from beet-sugar waste ash.

Carbon dioxide is passed through the lye (40° B.) at a temperature of 40–50° C. after the by-products have been partially removed therefrom. The carbon dioxide is used under pressure. At 39° B. the carbon dioxide is cut out and the potassium carbonate removed and the lye concentrated. The sodium bicarbonate in the lye is changed to sodium carbonate by heating. Carbon dioxide is again passed through the lye and more potassium carbonate is recovered.

Fr. 385139. Riviere, L. App. February 28, 1907; Pat. February 29, 1908; Pub. May 2, 1908.

Fr. Addition No. 8702. Riviere, L. App. March 28, 1907; Pat. March 30, 1908; Pub. June 12, 1908.

Fr. Addition No. 8908. Riviere, L. App. May 11, 1907; Pat. May 12, 1908; Pub. July 24, 1908.

Fr. Addition No. 9228. Riviere, L. App. July 1, 1907; Pat. July 8, 1908; Pub. September 17, 1908.

Title: Manufacture of depotassiumized molasses. Industrial use of these products.

This elaborate process includes treatment of vinasse for extraction of ammonia and its salts, glycerine, and a solid residue. The steps and apparatus outlined are as follows: (1) Apparatus for the preparation and regeneration of hydrofluosilicic acid (used to precipitate the potassium). (2) Apparatus for the precipitation of potassium and sodium, and, if needed, for the extraction of ammoniacal nitrogen before fermentation. (3) Fermentation and distillation of the depotassiumized molasses.

Ital. 135224. Vigano and Soldi. Pat. August 12, 1913.

Title: Obtaining ammonia and potash from molasses slop.

After the extraction of the alcohol from the molasses, the residual liquor is treated in a furnace, whereby a residue rich in potassium salts is obtained. This mass, after being drawn from the furnace in an incandescent state, is burned further "in pleno," cooled and crushed for the market. The nitrogen still contained in the furnace may be utilized by means of a circulation of air and water vapor with condensation of the resulting ammonium hydroxide, or the ammonia may be absorbed in sulphuric acid.

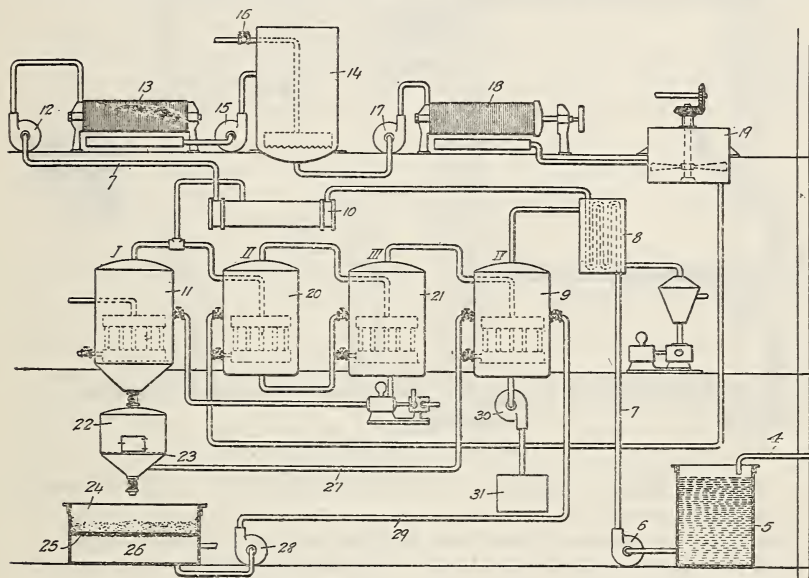


FIG. 43.—U. S. 1303916.

U. S. 1303916. Kermer and Faber. App. March 15, 1918; Pat. May 20, 1919.

Title: Method of reclaiming potassium salts, etc., from sugar refuse.

Waste water is heated to 185° F. and carbon dioxide added, then filtered. Sulphuric acid (0.2 per cent by weight) is added to precipitate the impurities and form soluble sulphates. The liquor is then evaporated until potassium sulphate begins to crystallize and subsequently dried to obtain nitrates in the residue.

U. S. 1376662. Whitaker, M. C., assignor to U. S. Industrial Alcohol Co. App. November 12, 1918; Pat. May 3, 1921.

Title: Process for obtaining alcohol and potassium compounds.

Cane molasses, diluted, is fermented in the presence of hydrofluosilicic acid in quantity insufficient to precipitate all the potassium salts in solution, using a yeast accustomed to the acid. After the alcohol is distilled sufficient acid is added to precipitate all the potassium salts. Sulphuric acid is added and the hydrofluosilicic acid may be recovered, while potassium sulphate may be purified by solution in water and recrystallizing.

U. S. 1400192. Whitaker, M. C., assignor to U. S. Industrial Alcohol Co. App. December 31, 1917; Pat. December 13, 1921.

Title: Process of obtaining potassium compounds.

Similar to the preceding patent, but adds the recovery of the hydrofluosilicic acid in the following manner: The acid is recovered from the potassium salt thereof by

the addition of another acid, distilling and absorbing the evolved gas in distillery waste, thus precipitating more potassium salts, introducing sufficient additional hydrofluosilicic acid to compensate for the loss thereof in the process, and then recovering the latter from the precipitate in the same manner.

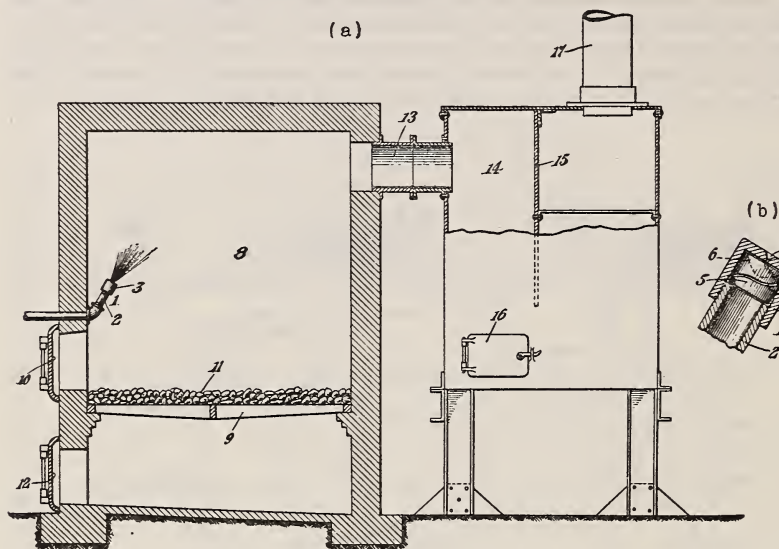


FIG. 44.—U. S. 1403160.

U. S. 1403160. Haner, C., assignor to U. S. Industrial Alcohol Co. App. February 19, 1920; Pat. January 10, 1922.

Title: Process of obtaining potash from distillery waste by burning the same.

Distillery slop or waste is sprayed into a combustion chamber to effect desiccation and obtain mixed potassium salts adapted for use as a fertilizer.

CROSS REFERENCES.

U. S. 1344850-1. Bassett. Group 7 (a).

Fr. 59268. Evrard. Group 7 (d).

U. S. 1145484. Huber and Poindexter. Group 4 (a).

Fr. 447138. Riviere, L. Group 7 (a).

U. S. 486647. Sternberg, L. Group 2 (a).

Ger. 78442. Sternberg, L. Group 2 (a).

7. GROUP VII.—RECOVERY OF MISCELLANEOUS PRODUCTS FROM MOLASSES, WASTE WATER, DISTILLERY SLOP, ETC.

(a) GLYCERINE, FATTY ACIDS, ESTERS, ETC.

Fr. 150999. Dehaut. Pat. September 7, 1882.

Title: Extraction of glycerine from vinasses from all sources. (Unpublished.)

Fr. 166948. Prenez. Pat. February 10, 1885.

Title: Extraction of glycerine from distillery vinasses. (Unpublished.)

Fr. 362654. Riviere, L. App. January 23, 1906; Pat. April 11, 1906; Pub. July 4, 1906.

Title: Apparatus and process for treating distillery by-products.

The apparatus and process described are for the treatment of distillery residues, such as those which are freed from the greater part of their potash by precipitation

(by means of 40-45 per cent hydrofluosilicic acid) on leaving the stills, and returned with as small a loss as possible, either to the diffusors or to the mash tuns, or, after utilizing the heat, to the drains. The process permits of recovery of the ammonia by distillation in autoclaves, evaporation and extraction of the residue for glycerine, and finally recovery of the alcohol.

Fr. 417950. Lederer, A. and E. App. June 8, 1910; Pat. September 15, 1910; Pub. November 25, 1910.

Fr. Addition No. 14315. Lederer, A. and E. App. May 24, 1911; Pat. August 22, 1911; Pub. November 17, 1911.

Title: Process of treatment of distillery vinasse.

Vinasses are repeatedly dried on an absorbent body, such as lignocellulose until a sufficient concentration has been built up. This is extracted with alcohol and the residue briquetted and treated in much the same manner as in the Meunier patents.

Br. 12207/11. Meunier, G. App. May 20, 1911; Acc. May 20, 1912.

Br. 13018/11. Meunier, G. App. May 30, 1911; Acc. May 30, 1912.

U. S. 1107175. Meunier, G. App. May 17, 1911; Pat. August 11, 1914.

Title: Method and apparatus for treating distillery wash.

In order to extract glycerine, succinic acid, and other useful products from vinasse, the latter is mixed with cellulose (preferably wood cellulose) which has been completely or partially carbonized with sulphuric acid, and the mixture is dried and extracted with suitable solvents, such as crude alcohol, aided by pressure or osmosis if necessary. The residue left after extraction may be molded into briquets and burnt, whereby alkaline salts or crude pearl ash is obtained; or, it may be subjected to dry distillation to obtain methyl alcohol, acetic acid, and other products. The coke from the distillation contains oxalic acid and cyanides, and after extraction of these by lixiviation, the residue may be used as a fuel or converted into a filtering material. The crude glycerine may be purified by further treatment with carbonized cellulose.

Ger. 253573. Guignard and Watrigant. Pat. May 27, 1911; Pub. Nov. 12, 1912.

Br. 12737/11. Guignard and Watrigant. App. May 26, 1911; Acc. December 21, 1911.

Title: Process of recovery of glycerine, fats, etc., from waste water.

Glycerol and fatty matters, or betaine, are extracted by evaporating the wash to dryness and extracting the residue with a mixture of absolute alcohol and a solvent, such as acetone or carbon tetrachloride, which is soluble in alcohol. Or, before it is dried, the wash is preferably treated for the removal of succinic, tartaric, citric, lactic, and other organic acids.

Fr. 445895. Watrigant Freres & Co. App. September 16, 1911; Pat. September 17, 1911; Pub. November 21, 1912.

Br. 21060/12. Watrigant Freres & Co. App. September 16, 1912; Acc. September 16, 1913.

Ger. 263354. Watrigant Freres & Co. Pat. September 10, 1912; Pub. August 6, 1913.

Title: Process of obtaining glycerine from waste water.

The residues are dried as completely as possible and extracted with various liquids in which the glycerine is insoluble under the conditions of the treatment, after which they are washed with hot amyl alcohol, and from this extract the dissolved glycerine separates on cooling. For instance, acetone may be used in the preliminary washing to dissolve potassium malate, while ethyl acetate is subsequently employed to dissolve the betaine. Ethyl acetate or a cold saturated solution of glycerine in amyl alcohol may be used to extract both potassium malate and betaine.

Fr. 447138. Riviere, L. App. October 18, 1911; Pat. October 19, 1912; Pub. December 23, 1912.

First Addition. Riviere, L. Pat. August 27, 1912.

Title: Rational treatment of wine and distillery vinasse.

Potash is recovered from the vinasse by either sodium silicofluoride or an alcoholic solution of sodium perchlorate. The residue is concentrated and treated with alcohol for the recovery of glycerine. The filtered solids from the alcohol treatment, after washing with dilute sodium carbonate, may be used as a fodder. The potash may be recovered from the glycerine instead of from the original vinasses. The fats present in the wine or vinasse are saponified by lime. The calcium soap, after separation from the liquid, is converted into soluble soaps or free fatty acids. The filtrate from the calcium soap is concentrated under reduced pressure, mixed with alcohol and filtered from protein matters. The cakes of the latter are treated in the filter-press with compressed air, carbon dioxide, or other gas, and afterwards dried for fodder. Alcohol is removed from the filtrate by distillation under reduced pressure, the residue is mixed with fine sand or similar material and the glycerol extracted with acetone.

Fr. 453073. Gouthiere, H., & Co., and Ducancel, P. App. March 25, 1912; Pat. March 26, 1913; Pub. May 30, 1913.

Title: Process of treatment of distillery vinasses for the extraction of glycerine and food material for cattle.

The glycerine is distilled with steam at reduced pressures from a mixture of concentrated vinasse, spent wash, and pulp. The process is intended particularly for grain fermentations.

U. S. 1344850. } Bassett, H. P. App. July 12, 1919; Pat. June 29, 1920.
U. S. 1344851. }

Title: Process of treating distiller's slop.

Boiling slops with lime converts the carbohydrates into salts of the hydroxyacids of the lactic acid series. Upon acidifying, the latter are decomposed with the formation of aldehydes and acids of the volatile fatty-acid series. Formic and acetic acids may be recovered by distillation. The solution is treated with a mixture of ferrous and ferric sulphate and made alkaline, precipitating basic iron salts of the fatty acids. The mixture is filtered and evaporated until about two-thirds of the potassium salts separate. The decanted liquid contains about 50 per cent glycerine, which is recovered by steam distillation.

U. S. 1357138. Bassett, H. P. App. November 3, 1919; Pat. October 26, 1920.

Title: Process of treating distiller's slop.

Distiller's slop is subjected to the action of the higher hydroxide of a metal which is capable of forming higher and lower hydroxides, and capable also of forming an insoluble basic salt with volatile fatty acids. The hydroxide is maintained in the higher state of oxidation by suitable oxidizing treatment, the liquid is acidified, and glycerol is separated from it.

U. S. 1396006. Backhaus and Haner, assignors to U. S. Industrial Alcohol Co. App. October 22, 1917; Pat. November 8, 1921.

Title: Method of treating distillery waste.

Distillery slop from the alcohol industry is treated with caustic alkali and the mixture dried to a clinker, which contains sodium salts of oxalic, acetic, formic, and butyric acids. This is heated with dilute sulphuric acid, and lime is added to precipitate calcium oxalate, which is filtered out. A further addition of sulphuric acid is made to the filtrate after its evaporation nearly to dryness and the liberated volatile acids are distilled in vacuo. A temperature of 175-250° C. is suitable for forming the clinker.

U. S. 1396007. Backhaus, A. A., assignor to U. S. Industrial Alcohol Co. App. October 22, 1917; Pat. November 8, 1921.

Title: Process of treating distillery waste.

In the process described in U. S. 1396006, carbon dioxide is passed into the filtrate, after precipitating calcium oxalate, in order to precipitate sodium bicarbonate, which is filtered out, incinerated, and heated with lime to regenerate sodium hydroxide.

U. S. 1396008. } Backhaus, A. A., assignor to U. S. Industrial Alcohol Co. App.
 U. S. 1396009. } October 22, 1917; Pat. November 8, 1921.
 U. S. 1396010. }

Title: Process of producing organic acids.

Distillery slop is concentrated to 25° B., 25 parts are mixed with 8 parts of sodium hydroxide and iron oxide, manganese dioxide, or cerium oxide, 1 part, and the

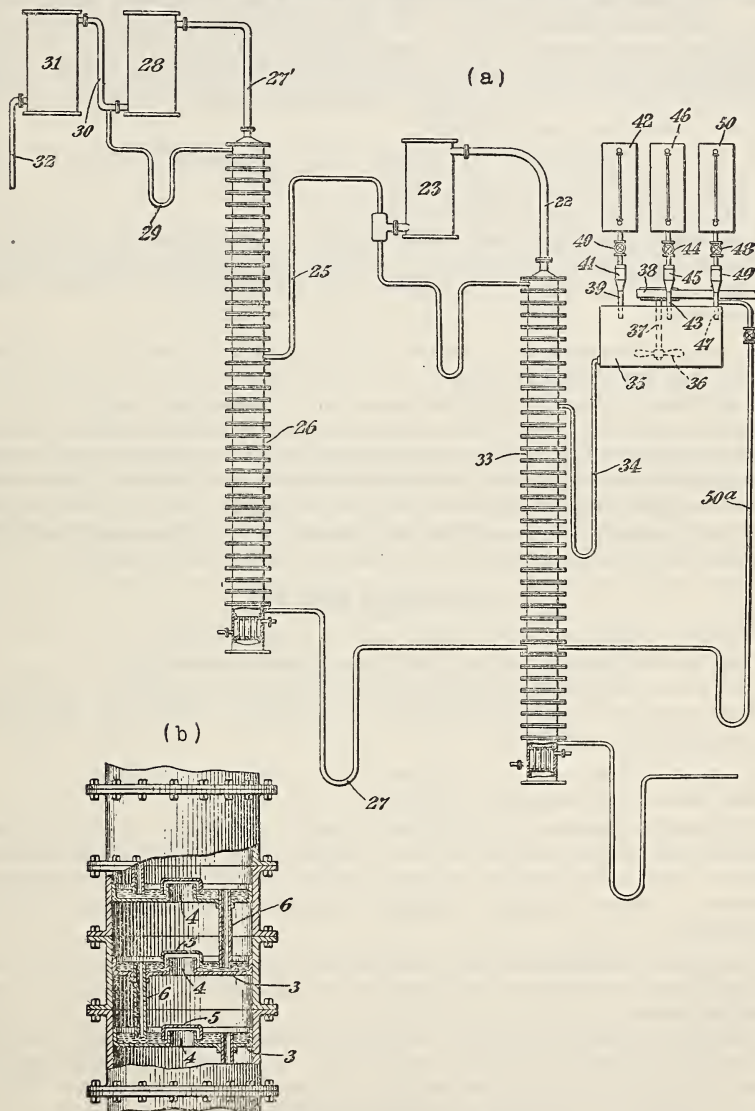


FIG. 45.—U. S. 1400852. (See p. 60.)

mixture is heated for 4-20 hours at 160-250° C. in order to produce sodium salts of organic acids in larger amount, more quickly, or at a lower temperature than without the use of the oxides as catalysts.

U. S. 1396368. Haner, C., assignor to U. S. Industrial Alcohol Co. App. October 22, 1917; Pat. November 8, 1921.

Title: Process of treating distillery waste.

Distillery waste is concentrated to a magma and then heated to 175–250° C. for 8–24 hours with sodium hydroxide to form a clinker rich in sodium salts of organic acids. This product is treated with oxalic acid to liberate the volatile organic acids and the latter are distilled in vacuo. The residue is treated with water and lime to precipitate calcium oxalate and the latter is filtered out and decomposed with sulphuric acid to obtain oxalic acid, which is crystallized after separation from calcium sulphate by filtration. The filtrate first obtained after adding lime may be concentrated to regenerate sodium hydroxide.

U. S. 1400852. Backhaus, A. A., assignor to U. S. Industrial Alcohol Co. App. May 23, 1919; Pat. December 20, 1921.

Title: Esters from fermented distillery waste.

Acid-fermented distillery waste containing organic acids is passed in countercurrent to gradually increasing concentrations of an alcohol; for example, methyl alcohol, in the presence of a catalyst, such as sulphuric acid, while continually distilling off the ester produced.

U. S. 1426457. Burghart, L. M., assignor to U. S. Industrial Alcohol Co. App. June 24, 1918; Pat. August 22, 1922.

Title: Process of recovering volatile organic acids from distillery waste.

A mixture of concentrated distillery slop and sodium hydroxide is dried and heated to form a clinker. This is heated and agitated with dilute sulphuric acid to effect neutralization, and the solution thus formed is filtered, after addition of lime to precipitate calcium oxalate. The filtrate, which contains sodium salts of volatile organic acids, such as formic, acetic, and butyric, is heated in a refluxing column still with sulphuric acid and methyl alcohol or other alcohol to obtain crude esters.

(b) FERTILIZERS, FEED, ETC.

Br. 6931/01. Fischel, I. App. April 2, 1901; Acc. May 24, 1901.

Title: Improvements in treating waste liquors of sugar manufacture.

A process is described to convert molasses, waste water, etc., into an easily portable form by absorbing in porous substances, such as sawdust, cotton waste, beet slices, etc., and drying.

U. S. 965521. Hoffman, E. A. App. June 24, 1909; Pat. July 26, 1910.

Title: Reducing distillery slop to dry feed.

By separating the slop as it is withdrawn from the distillery beer still into thick and thin slops, heating the thin slop to the boiling point, adding sufficient alkaline substance, such as lime, sodium carbonate, sodium hydroxide, potassium hydroxide, ammonia, or barium hydroxide to coagulate it, mixing with the thick slop and drying the residue.

U. S. 1134921. Sinsheimer, S. W. App. May 10, 1913; Pat. April 6, 1915.

Title: Concentrated product of nitrogen and potash and process of recovering and solidifying same.

A fertilizer is prepared from the waste liquor remaining after the sugar has been separated from molasses by heating to about 100° C. to precipitate part of the lime and sugar, filtering the hot liquid, and treating the filtrate with hot furnace gases, filtering out the precipitate thus formed, and concentrating and drying in a vacuum at a low temperature.

U. S. 1147767. Laszloffy, A. von. App. January 30, 1911; Pat. July 27, 1915.

Title: Manufacture of by-products from distillery slop.

Distillery slop is concentrated to a sirupy consistency and extracted with alcohol to precipitate the albumins, dextrans, gums, etc., which are used for stock food.

U. S. 1147768. Laszloffy, A. von. App. August 25, 1911; Pat. July 27, 1915.

U. S. 1147769. Laszloffy, A. von. App. February 26, 1912; Pat. July 27, 1915.

U. S. 1147770. Laszloffy, A. von. App. October 20, 1913; Pat. July 27, 1915.

Title: Manufacture of by-products from distillery slops.

Ether and alcohol are used as solvents for the recovery of acids, fats, and glycerol.

Br. 9270/15. Kaplan and Thompson. App. June 24, 1915; Acc. March 2, 1916.

Title: Fertilizer from molasses and vinasse.

Spent liquor is concentrated to 44° B., adding 30-50 per cent ground phosphate, guano, or bone meal, and treating with sufficient sulphuric acid to convert the phosphates to acid phosphate and potassium salts to sulphates.

U. S. 1212484. Herzka, E. App. October 24, 1916; Pat. January 16, 1917.

Title: Process of manufacturing fertilizer out of Steffen waste water, etc.

Steffen waste water or similar waste waters obtained in the extraction of sugar from beets are concentrated to 55° Brix, treated with sulphuric acid sufficient to neutralize the lime and other alkalies in the liquor, mixed with calcium superphosphate, sawdust, and nitrogenous animal wastes, and the mixture is dried.

(c) DYEING AND MORDANTING MATERIALS.

Br. 20851/02. Schrader, H. App. September 24, 1902; Acc. June 25, 1903.

Title: Improved process for obtaining and utilizing the organic acids contained in the residual liquors of molasses.

The process uses waste waters from the calcium, barium, or strontium desugarizing processes, or distiller's slop. The organic acids, such as betaine acids, are left in the residual liquors to use as a mordant in dyeing wool. The residual liquors or waste waters are concentrated to 1.4 specific gravity, then mixed warm with sulphuric acid (92 per cent) to neutrality. Potassium sulphate is separated from solution, and the residual sulphuric acid is separated by precipitation after dilution. The filtrate is decolorized. It finally has a specific gravity of 1.1-1.2, and contains 24 per cent of acid of a molecular weight of 135. This liquor is used in combination with metallic mordants for treating and dyeing wool.

Ger. 141564. Schrader, H. Pat. February 1, 1902; Pub. May 13, 1903.

U. S. 761412. Schrader, H. App. July 1, 1902; Pat. May 31, 1904.

U. S. 735599. Schrader, H. App. March 19, 1903; Pat. August 4, 1903.

Fr. 328160. Schrader, H. App. December 1, 1902; Pat. December 1, 1903; Pub. February 8, 1904.

Title: Process of manufacture of organic acids contained in molasses waste water.

In addition to the information given in Br. 20851/02, the use of barium carbonate to neutralize the sulphuric acid is added, and the use of animal and vegetable charcoal to decolorize.

Ger. 167122. Wichardt, O. Pat. April 3, 1904; Pub. December 30, 1905.

Title: Process for the manufacture of green to blue dyestuffs containing molybdenum.

Ammonium molybdate reacts on molasses, waste water, or beet juices, when heated. Acid is added and the precipitation of the dyestuff is completed by the addition of alcohol. The intensity of the color is determined by the quantity of sulphuric acid added and varies from light green to dark blue. The dyes are for dyeing wool and silk, and for use as pigments.

(d) MISCELLANEOUS PRODUCTS—ALKALIES, PHOSPHATES, ETC.

Fr. 59268. Evrard. Pat. July 17, 1863.

Title: Preparation of various products from molasses vinasses and analogous materials.

Vinasses are concentrated to 40° B., potassium nitrate separates out and is leached. The mother liquor is introduced into a cylindrical retort and dry distilled, yielding

wood spirit and nitrogenous products, especially ammonium carbonate. These products are then led into a cylinder containing alkaline char from a previous distillation and are converted to cyanides, potash and soda. If finely divided iron be added to the char before calcination, potassium ferrocyanide is formed.

Ger. 28838. Propfe, H. Pat. March 18, 1884; Pub. August 25, 1884.

Title: Process of recovery of tar products and alkalies by distillation of desugarized wastes of molasses desugarizing with hard coal tar or tar oils.

Peat is added to lyes, then tar. The mixture is distilled in iron or clay retorts. The gases are driven over by steam, and at the end the residue is in the form of a porous coke. If it is desired to recover the alkalies, lime is added before calcination.

Fr. 396288. Verbieese, F., and Darras-Verbieese, E. App. January 21, 1908; Pat. January 22, 1909; Pub. April 6, 1909.

Fr. Addition No. 10370. Verbieese, F., and Darras-Verbieese, E. App. January 19, 1909; Pat. April 7, 1909; Pub. June 24, 1909.

Title: Process of treating vinasses of distilleries.

The vinasses are clarified with iron chloride and aluminum sulphate. Lime is added as a milk to precipitate the clarifying agents, matter in suspension, phosphates, a large part of the nitrogen, various acids, and organic matter. This precipitate is filtered on a press, the press cakes dried and used as fertilizer. The excess of lime is carbonated. The powdered cake from beet vinasses contains about 5 per cent phosphate and 5 per cent nitrogen.

Fr. 485597. Dupont, L. App. July 25, 1916; Pat. October 30, 1917; Pub. January 23, 1918.

Title: Process of treating beet and cane molasses, and vinasses therefrom, to obtain ammonia, amines, acids, and other organic compounds.

The initial materials are treated at about 300° C. with caustic alkali to expel ammonia, which is recovered by known means. The residues from this treatment afford solid salts or organic acids, especially oxalic and acetic, which are separated by difference of solubility. Certain volatile organic compounds are obtained with the ammonia, such as amines, phenolic and alcoholic bodies.

U. S. 1342737. Bradford and Broadhead. App. September 16, 1918; Pat. June 8, 1920.

Title: Method of recovering by-products from waste liquors.

Steffen waste water is concentrated to about 30° B. and then subjected to the action of an electric current of sufficient intensity to produce an arc in the liquor. This effects evaporation with electrolytic dissociation and then complete combustion. The gaseous products of evaporation and combustion are collected and cooled to recover methylamine, cyanogen, methyl chloride, and the residue is leached to obtain the potassium chloride.

CROSS REFERENCES.

Fr. 442923. Gouthiere, H., & Co., and Ducancel, P. Group 2 (a).

Fr. 353970. Guerrero, W. de S. Group 2 (b).

Br. 17347/87. Meyer, E. Group 2 (a).

Ger. 43345. Meyer, E. Group 2 (a).

U. S. 396705. Meyer, E. Group 2 (a).

Fr. 385139. Riviere, L. Group 6.

Ger. 215531. Societe Anonyme de Recuperation de Produits Chimiques "Procede Effront." Group 2 (a).

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